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# COAL SAMPLING AND ANALYSIS:

## Methods and Models

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The report provides information on coal sampling and analysis (CSD) techniques and procedures and presents a statistical model for estimating SO2 emissions. (New Source Performance Standards for large coal-fired boilers and certain State Implementation Plans require operators to monitor SO2 flue gas emissions: as an alternative to stack gas monitoring, sampling and analysis of feed coal has been proposed to estimate SO2 emissions.) In particular, this study assesses the various coal sampling techniques and equipment, the various sample preparation and analytic methods, and common practices for CSA. It describes the variables associated with the prediction of SO2 emissions from CSA data; e.g., sulfur retention, variability, measuring errors, and auto-correlation. Finally, it presents a time series model for predicting emissions which takes into consideration the correlation of the sulfur content of the coal, the measuring errors, and the sampling procedures for coal collection. The model is used to fit 53 data sets with little evidence of non-fit.

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| Sampling | | | 14B | |
| Analyzing | | | | |
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# ABSTRACT

New Source Performance Standards for large coal-fired boilers and certain State Implementation Plans require operators to monitor $SO_2$ flue gas emissions. As an alternative to stack gas monitoring, sampling and analysis of feed coal has been proposed to estimate $SO_2$ emissions. This report provides information on coal sampling and analysis (CSA) techniques and procedures and presents a statistical model for estimating $SO_2$ emissions. In particular, this study assesses the various coal sampling techniques and equipment, the various sample preparation and analytic methods, and common practices for coal sampling and analyses. It describes the variables associated with the prediction of $SO_2$ emissions from CSA data such as sulfur retention, variability, measuring errors, and auto-correlation. Finally, it presents a time series model for predicting emissions which takes into consideration the correlation of the sulfur content of the coal, the measuring errors, and the sampling procedures for coal collection. The model is used to fit 53 data sets with little evidence of non-fit.

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# SECTION 1

# INTRODUCTION

Coal sampling, whether performed manually or automatically, must extract a quantity of coal much smaller than the original lot for laboratory analysis. The sample, to be representative, must have the same characteristic qualities and constituents as the entire coal lot. However, inherent variability in the coal parameters and the imprecision of the entire measurement process make sampling a rather technical subject. This report addresses the methodology, equipment, and modeling of the coal sampling and analysis procedures.

Section One of the report provides an introduction concerning the subject of the report and the contents of each section. Conclusions are provided in Section Two.

Section Three discusses the various sampling techniques and equipment. The most widely regarded standards for sample collection and preparation were established by the American Society for Testing and Materials (ASTM). Their Method D 2234 details the minimum number and weight of the increments and the size of the gross sample needed to provide a stated level of precision. ASTM also evaluates the conditions under which an increment is collected.

The collection of coal samples by manual and mechanical means is next reviewed. Most mechanical systems are based on the ASTM standards for cutter speed and size. The specifications and operation of several augers (for manual sampling) and cutters (for mechanical sampling) are given.

Section Four discusses the industry's sample preparation and analytic techniques that are currently in use. The primary sample may weigh as much as several hundred pounds whereas the laboratory requires but 50 to 100 grams.* Hence sample preparation techniques such as air-drying, crushing, sample division, pulverizing, and mixing are employed. Procedures and equipment to perform each of these steps are described with particular emphasis on identifying factors which may increase the variance or reduce the representativeness of the resulting sample.

Next, the analytical methods used to determine moisture, total sulfur, ash content, and calorific value (heat content) are described. The appropriate ASTM and International Organization for Standardization (ISO) methods are given and equipment available to perform the analyses are described. Quality control measures to ensure the reliability of the analytic methods and the precision of the data are suggested.

Section Five discusses the potential for sulfur loss as the coal moves from bunkers through the pulverizers and the boiler. Nuggets of pyritic sulfur may be removed by the pulverizers. Mass balance relationships are difficult to perform; they do suggest a sulfur retention in the bottom ash and fly ash as well as some $SO_3$ mixed in with the $SO_2$ in the stack emissions. Several sulfur retention guidelines for EPA's consideration are offered.

Section Six identifies common coal sampling and analysis techniques used by coal-fired utilities to evaluate their fuel. This information is important to the study because it indicates how closely current industry practices match the proposed EPA Reference Method 19A requirements.

Section Seven discusses in a relatively non-technical manner the various coal sulfur parameters. Daily $SO_2$ stack emissions are estimated on the basis of daily composited samples of coal. The accuracy and precision of these estimates is of vital concern to EPA. This section discusses the role of time series data analysis, variances, and auto-correlation on the estimation of current and the prediction of future emissions. The role played by the measuring process will be explicated. The importance of the various coal sulfur parameters vis-a-vis compliance with emission standards is described. Techniques which may be used to reduce variability and/or correlation are included.

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*To convert from English units to metric units, see the conversion table on Page 155.

Section Eight describes a theoretical statistical model for estimating and predicting emissions data from coal sampling and analysis data. It is assumed that as-bunkered coal can be represented as Q discrete packets of homogeneous material per day. The computer implementation is based on Q = 1,008,000. The cutter passing through the stream collects L of these packets per increment (computer implementation has L = 24). The daily composite C is made up of N increments (ASTM recommends N = 35 for some boilers).

The actual data X is made up of the true value C plus a possible measurement error. The model assumes that X can be described using a fixed time series model. Estimation for the parameters of this model are provided.

Section Nine considers the measuring error in detail. The data X will be more representative of the actual coal C if the measurement error in the sampling and analysis procedures is minimized. Considerations of sampling error, sampling location, sample lot size, sampling system performance, sample preparation, and sample analysis are discussed; some elements of a quality control program to minimize these terms are given.

Section Ten discusses bias. Systematic error, or bias, in a sampling system will consistently misrepresent the actual coal in the same way. This can lead to inaccurate (and probably misleading) analytical results. To prevent this, a sampling system can be bias tested. The proposed ASTM stopped belt procedure and one based on the time series model presented earlier in Section Eight are offered as possible bias tests. Using the known size-frequency bias for coal from the EPRI Coal Cleaning Test Facility and the known sulfur size-frequency distribution for two different coals, the simulated sulfur bias was found to be negligible.

Tests of the model are presented in Section Eleven. The statistical model was verified by fitting 53 data sets. Though the model is simple and general purpose, there was no real indication of lack of fit. Splitting the data into pieces produced consistent estimates for the model parameters. Long term stationarity (i.e., mean, variance, etc., constant over time) does not appear to hold.

Last, Section Twelve, describes the beginning of a causal model between emission data and coal and boiler characteristics.

It is our conclusion that additional work will need to be done to refine the models in order to obtain better fits to actual data. This report represents only a first approximation to the true situation and should be read in that light.

# SECTION 2

# CONCLUSIONS AND RECOMMENDATIONS

Coal sampling and analysis (CSA) procedures cannot guarantee "correct" results. Inherent coal variability and the representativeness of the sampling and preparation procedures lead us to conclude that the resulting 50-100 grams of coal per day analyzed in the laboratory may not have coal parameters exactly equal to the daily average of the coal. The laboratory results rely on falible humans which may introduce additional inaccuracies. Thus, any discussion of CSA must address the statistical issues of estimating the true emissions from the scattered sulfur data.

This study presents a theoretical model for coal sulfur data involving time series modeling. Extensive verification of the model using real coal data does not suggest any widespread lack of fit. On the contrary, the model seems to do a very credible job in fitting the data. The model can be used by EPA to evaluate the impact of various stack emissions standards and/or averaging periods on the mean level of compliance coal. Alternately, it can be used by EPA to offer guidelines to the industry as to how it could meet proposed stack emission standards.

## SECTION 3

## ASSESSMENT AND STATISTICAL EVALUATION OF COAL SAMPLING TECHNIQUES AND EQUIPMENT

The purpose of this section is to document industry accepted coal sampling methods and define the specific sampling principles involved. The objectives of coal sampling are discussed, including the procedures and rationale for collecting a representative sample. The development of accepted coal sampling guidelines are presented. Sampling equipment is evaluated according to its effectiveness in obtaining a representative sample according to accepted standard methods. A discussion of sampling considerations and objectives include suggested procedures to achieve the level of accuracy needed to meet specific standards. Coal sampling is presented as a means of predicting $SO_2$ emission levels.

### 3.1 OBJECTIVES OF COAL SAMPLING

The sampling of coal, whether performed manually or automatically, must extract a quantity of coal much smaller than the original lot but with proportionately the same characteristic qualities and quantities present in the entire lot. It has long been realized that the properties in coal are not distributed uniformly. The variability of coal makes it difficult to collect a sample that is representative of a large mass of coal. For instance, grab samples of coal from the same source may show different analytical values if tested in different laboratories or by different technicians in the same laboratory.(1)

Besides coal's inherent variability, other factors such as how the coal was handled, or how the samples were obtained, affect the collection of a representative sample. The coal may have become segregated during loading, transport, or unloading operations so that the particles are grouped together by size. When samples are taken from a stationary source, such as a coal storage pile or railroad car, it is difficult to obtain an accurate sample because the material located in the center of the pile will be inaccessible when conventional sampling techniques are used. Generally, samples taken around the pile will be limited by the depth of penetration of the sampling device into the stationary source. Similarly, when samples are taken from a moving stream, such as a conveyor, they should be taken from the entire width of the belt to avoid biasing the sample.

To be effective, a sampling plan must employ measures to reduce the effect of segregated particles, minimize the effect of the variability of the coal properties, and identify any mechanical bias due to the sampling method. Sampling material from a conveyor or a chute through which the coal is flowing provides access to a cross-section cut of the entire stream. This cross-sectional cut will provide a characteristic sample even though the vertical distribution of material on the conveyor may be segregated by particle size.

Mechanical sampling bias may be reduced by the use of automatic equipment which is not dependent on human discretion for operation. These systems are generally elaborate and have been designed for a specific plant's application. Manual sampling methods can be used, but care must be taken to ensure that the sampling technique has been consistently applied.

Sampling personnel must consider the variability between discrete units of coal when attempting to collect a sample which is representative of a specific lot. As an example, a 10,000-ton lot of coal may be made up of 100 discrete units or railroad cars of 100 tons each. If the variability between railroad cars is high because of differences in loading procedures and coal characteristics, a composite made up of one increment collected from every tenth car may be insufficient to represent the entire 10,000-ton lot. In this case, increasing the number of increments or increasing the number of railroad cars sampled, will produce a composite which better characterizes the entire lot. On the other hand, if the variability is expected to be relatively low, then sampling from each car may be an extensive effort with little or no extra benefit.

## 3.2 SAMPLING GUIDELINES

With all these factors contributing to the inaccuracy of coal sampling, there has been concern over the reliability of coal analysis data. Inaccurate data, whether due to an error in sampling, poor analytical techniques, or some other factor, can result in data misuse. Because of this, there are many opinions concerning guidelines for sampling coal and the establishment of standard methods.

As early as 1914, the United States Bureau of Mines (USBM) presented a procedures, written by A. C. Fieldner, for sampling coal in a mine.(2). The Bureau also developed guidelines for representative sampling for analysis(3). The author, G. S. Pope, points out that it is difficult to obtain a representative sample from a stationary source such as a railcar or storage pile. He further states that the only representative sample that can be collected from a heterogeneous mixture of coal is one from a moving stream of coal as it is being loaded or unloaded. Pope also offers general directions for the number, size, and frequency of sample increments.

Pope states that the conclusion reached by tests run by the USBM indicates that the gross sample should not be less than 1,000 pounds. The increments should be systematically spaced so that the entire quantity is represented in the gross sample. Pope suggests increments of 10 to 30 pounds, but he points out that increment size is governed by the size and weight of the largest pieces of coal; increments of greater than 30 pounds may be necessary.

A more specific guideline was provided by Bertrand A. Landry in another USBM publication(1). The author applies the theory of random sampling to coal sampling methods. Specifically, Landry used random sampling theories to determine the optimum number of increments to be collected to yield a gross sample of definable quality. The determination of the number of increments is based on the desired representativeness of the gross sample and assumes that the variability of the increments is known. Landry presents an equation for this determination:

$$N = \frac{S_W^2}{S_{(N)W}^2}$$

which states that N, the sufficient number of increments, can be found by dividing the variability of the increments of weight W, $S_W^2$, by the variability of gross samples of accepted representativeness, $S_{(N)W}^2$. This is based on the premise of the random chance of choosing each increment, and on all increments being the same weight. The total weight of coal being sampled is not considered in determining N for random sampling. The weight of each increment is assumed to be constant, but no specific weight is suggested. Landry then determines that systematic sampling will produce a sample as representative as random sampling when the total lot size is relatively large.

The U.S. Steel Corporation has also written directions for providing representative samples for analysis(4). They advise removing a full cross sectional cut from a moving stream or stopped belt conveyor, but they discourage sampling from railroad cars and other stationary sources. U.S. Steel recommends the collection of one increment every 30 minutes for moving streams and provides guidelines for gross sample requirements using different sampling schemes. A minimum of 1,000 pounds is specified for a gross sample. A sample increment weight of six to eight pounds is recommended when samples are collected from chutes or stationary sources. When sampling from a stopped belt a much larger increment is required. The cross-sectional cut should be a minimum of one foot in length.

To achieve a more uniform, systematic method for collecting coal samples, the USBM published a paper for government employees to use as a guideline in obtaining representative samples(5). This paper suggests systematic collection of a large number of increments of equal weight. The exact number and size of increments is dependent on the maximum size of the coal. In the case of a coal whose top size is 3/4 inches, 50 increments of 10 pounds each are required. For larger sized coal, the number of increments remains at 50, but the increment weight is increased. For example, a coal whose top size is 8 inches requires increment weights of 20 pounds each. The USBM recommends a full cross-stream ~~cut be taken from~~ a moving stream. They discourage top sampling from railroad cars or barges. If, as a last resort, top sampling is used, increments should be taken from throughout the car or barge.

All of these references give general recommendations for collecting a gross sample of coal, but none is an industry accepted standard method. The most widely regarded standards were established by the American Society for Testing and Materials (ASTM)(6). The ASTM issued recommended procedures for a variety of sampling situations. The ASTM Method D 2234 details the minimum number and weight of increments and the amount of the gross sample needed to provide a stated level of precision.

The ASTM method of determining the number and weight of increments, similar to USBM guidelines(5). The number of increments needed to achieve a required degree of precision is calculated using a relationship between several sampling variances and the increment number such that:

$$N = (S_s^2 + S_r^2/W)/(S_G^2 - S_{da}^2/P)$$

where:

$N$ = number of increments,

$W$ = weight of each increment,

$S_s^2$ = segregation variance,

$S_r^2$ = random variance of a 1 pound increment,

$S_G^2$ = variance of 1 gross sample,

$S_{da}^2$ = variance of division and analysis, and

$P$ = number of analysis samples.

The ASTM method used assumptions from a specific sampling situation to calculate that a minimum of 15 increments was necessary for each gross sample representative of a lot of 1,000 tons of mechanically cleaned coal. In the situation where the lot size is expected to be more than 1,000 tons, one gross sample can be collected to represent the higher tonnage. However, more increments are required. The number of increments can be expressed as a ratio of the expected lot size to the 1,000 ton lot size, as follows:

$$N_2 = N_1 \sqrt{\frac{\text{total lot size (tons)}}{\text{1,000 tons}}}$$

where:

$N_1$ = 15 increments
$N_2$ = number of increments required.

The weight of each increment is based on top size to minimize bias due to particle size. Generally, the larger the particle size present in the lot of coal, the heavier the increment should be. For a top size of 5/8 inch, ASTM specifies 2-pound increments. A top size of 2 inches requires 6-pound increments, while a 6-inch coal requires 15-pound increments.

The ASTM also evaluates the conditions under which an increment is collected, such as: stopped-belt cut, full-stream cut, part-stream cut, and stationary sampling. ASTM points out that an automatic or mechanical sampling method, without human discretion, is more precise than manual sampling. They classify each condition under which an increment is collected. The highest classification, or most precise method, is the stopped-belt cut removed by a mechanical cutter with increments spaced systematically. The stopped-belt technique allows all of the particles in a cross-section of the belt to be collected, eliminating segregation due to particle size. ASTM specifications restrict the cutter speed to 18 in./sec and indicates the minimum cutter width (size of the opening) of 2 1/2 to 3 times the topsize of the coal.

ASTM also considered the ratio of maximum particle size and the large variation of ash content among coal pieces of different size. Ash content was used because it was believed to be the most sensitive measure of variations in coal quality. ASTM specifies that no less than the minimum increment weight be collected so as to reduce the bias due to particle size and ash content.

These factors were discussed by Orning, who studied the distributions of particle sizes, ash contents, and their effect on the precision of sampling(7). Orning estimated the influence of various sizes and ash contents of coal on the variability of ash content in duplicate coal samples. He found that the sampling variance is more dependent on particle size and the possible rejection of large pieces of coal than on increment weight. Orning would permit a lesser number of smaller increments when coal of small top size is being sampled.

The theory of sampling based on the ASTM sampling principles was also discussed by Visman and Aresco.(8) The theory provides estimates of the variance of a coal, then provides the minimum number and weight of increments needed to attain a desired level of precision.

Another view is taken by Bertholf(9) who theorizes that a large number of almost weightless increments should be taken. The minimum increment weight proposed by Bertholf's theories is impractical based on the mechanical limitations of samplers. Therefore, he proposes the collection of a primary increment, larger than needed, which is then resampled, resulting in a secondary increment closer to the minimum weight requirements. This method would apply especially to mechanical coal samplers which take primary increments large enough to meet ASTM requirements each time the device is activated. The quantity of each primary increment collected is a function of the amount of material in the moving stream and can exceed several hundred pounds when these ASTM specifications are observed. This bulk of material far exceeds the quantity needed for analytical purposes and is much larger than the ASTM minimum increment weight of 6 pounds for 2-inch topsize material. In a study of mechanical coal sampling systems, Bertholf showed that large representative increments could be collected and reduced to smaller samples without a loss of precision(10).

Coryell et al. tested a different type of mechanical sampler which took primary increments of approximately 50 pounds and reduced each to a secondary increment of 3 pounds. These tests showed that the desired ASTM precision could be achieved by increasing the size of the primary increments while decreasing the number of increments(11).

It is generally accepted within these various guidelines that the number of sample increments should be based on lot size and sampling variance with increment weight being determined by considering maximum particle size. However, a different approach is suggested by Gy(12,13). Gy bases his methods on the theory that coal characteristics are not randomly distributed but are autocorrelated. Using the autocorrelation theories, Gy estimates a sample size based on particle size and shape. The minimum number of increments would then be determined from the estimated total sample size, the flowrate of the stream, and the width and velocity of the cutler. Other guidelines estimate a total sample size based on lot sizes, and the minimum number of increments is based on variance and the required precision. The theory that coal is autocorrelated and not randomly distributed is also addressed by Gould and Visman(14).

Another recommendation that Gy makes concerns cutter width and speed. Gy conducted experiments to determine the critical value of these two parameters. The critical value for the cutter width was determined to be three times the particle diameter, similar to the ASTM requirements. The critical value of the cutter speed was determined to be .6 m/s, a value slightly higher than the ASTM recommendation.

Table 1 summarizes the sampling guidelines and methods presented in this section.

## 3.3 SAMPLING EQUIPMENT

### 3.3.1 Manual Sampling

Although the recommended procedure for collecting a coal sample is with an automatic sampling system, manual sampling is sometimes the only alternative. Manual samples can be taken from a stationary source or, more reliably, from a moving stream. In this discussion of coal sampling equipment, some commonly used hardware will be described.

In stationary source sampling, such as from railroad cars or storage piles, shovels, buckets, probes, augers, and other instruments can be used. The simplest, and perhaps most inaccurate means would be sampling with shovels or scoops from a stationary source. Although discouraged, there are several guidelines for this type of sampling (2,4,5). All of them recommend taking several increments from different points and at a

TABLE 1. SUMMARY OF SAMPLING GUIDELINES

| Author | Recommended Method | Number of Increments | Size of Increments | Gross Sample | Recommendations |
|---|---|---|---|---|---|
| USBM/G.S. Pope | From a moving stream | Spaced systematically over entire lot | 10-30 pounds | 1,000 pounds | Larger increments result in more accurate sample |
| USBM/B.A. Landry | From a moving stream | $N = \frac{s^2 W}{S^2 (N)W}$ | All increments must be same size | Not considered | Sampling is based on variability of coal and desired precision |
| U.S. Steel | Full cross-section, stopped belt | One every 30 minutes | 6-8 pounds | 1,000 pounds | Orderly spacing of increments to represent entire lot |
| USBM/N.H. Snyder | Full cross-section, moving stream | 50 | 10-30 pounds | 500-1,500 pounds | Increment size dependent on coal top size |
| ASTM | Full cross-section, stopped belt | Minimum of 15 | 2-15 pounds | Dependent on increment number and size | Increment size dependent on coal top size |
| A.A. Orning | Not specified | Number and size of increments depends on variability of coal's size and properties. | | | |
| W. M. Bertholf | Not specified | Minimum number based on coal's variability | Not specified | Not specified | Proposes collection of a primary increment, which is then resampled |
| Dr. P. Gy | Mechanical method | 40 to 50, based on estimated sample size | Dependent on speed and width of cutter | Dependent on size and shape of particles | Recommends a mechanical sampler with a large cutter width |

consistent depth. This method will not always produce a representative sample of the entire lot because only the uppermost particles in the storage pile or transport vehicle are accessible to the sampling device.

An auger can be used to obtain a core sample of consistent depth into the pile. As an example, the Holmes Model 13 has a 30-inch auger (Fig 3-1) with a 4-inch diameter which limits it to coal with a topsize of 1.25 inches or less. Larger truck-mounted, boom-operated power augers are available and are commonly used to collect feedstock samples from railroad cars. The augering technique may not accurately represent fine particles because they tend to fall out of the sampler prior to discharge into the sample collection container. Although the use of an auger allows deeper penetration into a pile than shoveling, quite often the inner portions of the pile remain unsampled.

Preiser/MINECO offers slotted probes of varying lengths and diameters (Fig 3-2). These devices may be inserted into a pile but are limited to materials with a nominal topsize of less than 0.5 inch.

Samples taken from a moving stream by manual techniques are more precise than samples from a stationary source, but they are subject to human error. The human factor involved in accurately repeating the cutting process adds to the sampling error. For sampling material from a falling stream, the simplest devices include shovels, scoops, and buckets. The shovel or similar device should have raised sides and capacity sufficient to hold a cross-sectional cut without overflowing. Using the ASTM guidelines of sampling at a rate of 18 in./sec or less and using a cutter which has an opening at least 2.5 to 3 times the topsize of the coal, collecting a sample from conveyor moving coal at a rate of 500 tons/hr. would yield a sample weighing 200 to 300 pounds.

An increment this size would be very difficult to handle manually; therefore, a partial cross-stream cut should be considered. Although less precise then the full cross-stream procedure, multiple partial cross-sectional cuts should be taken at different locations until the

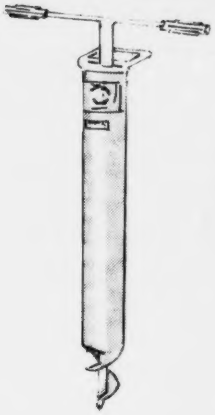


Figure 3-1. Holmes sample probe, Auger style.

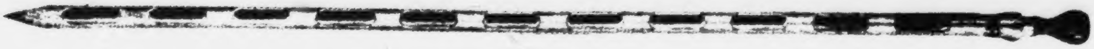


Figure 3-2. Sample probe, aluminum, 66" long, 11 openings, 1 3/8" O.D.

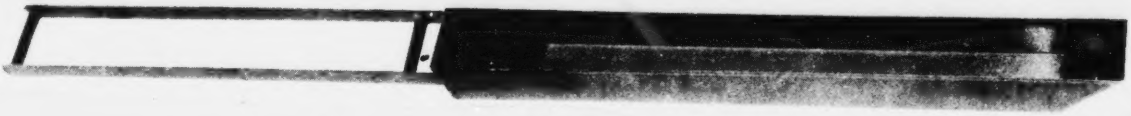


Figure 3-3. Preiser hand sampler.

entire stream has been sampled. Extreme care must be used when taking manual samples are taken from a moving stream, because the collection container generally fills very rapidly and may be quickly swept from the operator's hands. Samplers should use a device that has a secure handle and that can be rested on a solid object such as a chute access door frame or a railing along a conveyor transfer point.

Preiser/MINECO distributes a hand sampler which is a good tool for obtaining a cut or increment from a falling stream (Fig 3-3). The pan or trough section comes in varying sizes as large as 8 inches wide, 8 inches deep, and 48 inches long. Depending upon the flowrate of the stream and the topsize of the particles, a full cross-sectional cut can be taken with the larger samplers that meets the ASTM cutter opening size specifications. As with all manual sampling devices, human discretion is involved, which can affect the precision between successive increments.

Manual sampling from a moving belt may, at some facilities, pose a serious safety problem. If plant operations allow, the conveyor should be stopped and a complete cross-section taken. The width of the cut should be at least 2.5 to 3 times the size of the largest particle. The sides of the cut should be parallel. Care must be taken to remove all particles within the measured cross-section. A hoe or similar tool can be used to remove the material along with a brush for the finer particles. This stop belt method is the basis by which ASTM evaluates all automatic and manual methods for sampling of moving streams for bias.

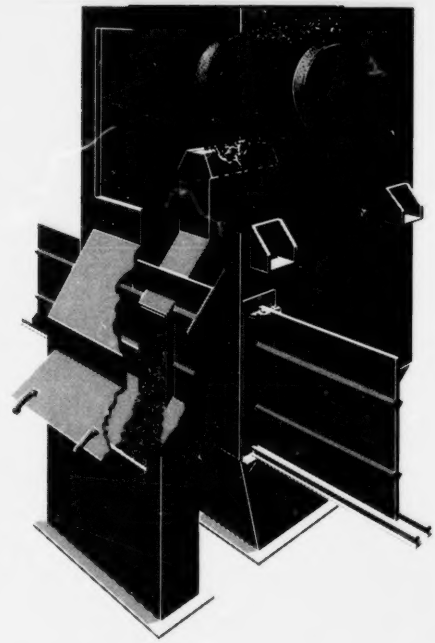
Manual sampling techniques, although subject to errors associated with human discretion, may be used to effectively collect samples of definable quality. Automatic sampling systems, once designed, installed, and tested for a specific plant application, however, will routinely produce samples which are cost-effective and, in terms of quality, generally exceed those collected by manual methods.

### 3.3.2 Mechanical Sampling

The collection of coal samples by mechanical means is considered a much more precise and representative method than manual sampling. The ASTM bases this assumption on the fact that an automatic sampler will not select an increment on a discretionary basis. There is no human element involved.

Most manufacturers of mechanical sampling systems have used designs based on ASTM standards(15,16). Several types are now in use including full cross-stream cutters, rotating scoops, augers, and rotary arm samplers. The reliability and accuracy of different automatic systems is difficult to quantify and is a function of the installation of the equipment, the physical characteristics of the material to be sampled, and the operating techniques used by the facility personnel. As previously mentioned, systems of this type as generally evaluated based upon a comparison with the ASTM stopped belt technique (ASTM D-2234-76, Appendix A). A standardized bias test does not exist, and in many cases sampling systems manufacturers have developed their own tests to evaluate the performance at the time the system is installed. A procedure proposed by Orning(7) consisted of compositing replicate gross samples from interleaved increments and determining the variance. This procedure was employed by Aresco and Orning(17) in a U.S. Bureau of Mines study.

The cross-stream cutter type system is widely accepted as providing a representative primary sample increment. The Ramsey/Galigher Engineering Company designed a cutter sampler to meet ASTM specification D-2234-76 (Figure 3-4). A full cross-stream cut is taken as the coal discharges from a conveyor head pulley. The cutter starts at one side of the belt discharge and cuts through to the other side so that the entire cross-section is represented. The cutter speed and opening can be adjusted to meet ASTM requirements. The Ramsey/Galigher sample cutters are designed for flow rates of up to 10,000 tph.



### CONDENSED SPECIFICATIONS

Model B-1800 and C-1800 Primary Sampling Machine:

| | |
|---|---|
| Capacity: | to 10,000 tph |
| Particle Size: | up to 12" top size |
| Cutter Stroke: | 72" to 96" or as req'd. |
| Cutter Type: | "C" 48'-96" |
| Drive Speed: | Hydraulic: to 30 in./sec.<br>Electric Chain Drive: 6, 12 or 18 in./sec. |

Figure 3-4. Ramsey/Galigher primary sampling machine.

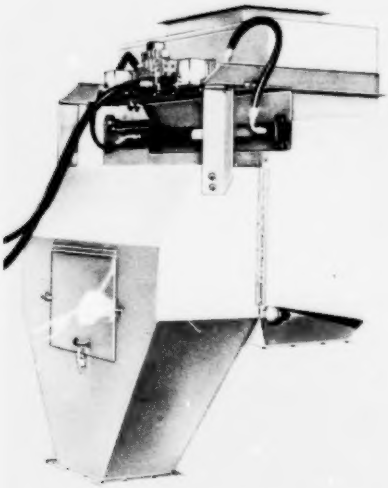
Similar systems are produced by the Denver Equipment Division (Figure 3-5). The Denver model samples a stream as it falls through a chute. The cutter speed and opening are adjustable, and a full cross-sectional cut is obtained.

The Quality Control Equipment Company also has a cross-cut sampler designed for chutes. However, the maximum particle size is set at two inches (Figure 3-6).

The McNally Belt Sampler takes the discharge from a belt and passes it through a chute section where the sample cutter removes a full cross-section (Figure 3-7). The McNally design is consistent with ASTM specifications. The cutter opening can be set to three times the size of the largest particle. For higher tonnages, McNally designed a travelling plate sampler (Figure 3-8). The cutter is driven through the stream of coal falling through a chute. The plate-sampler is suitable for large size material, and the speed of the cut can be adapted to ASTM specifications.

A traversing sampler which obtains a full cross-sectional cut from coal flowing in a chute has been developed by Harrison R. Cooper Systems, Inc. (Figure 3-9). The sampler maintains a constant cutter speed of 18 in./sec and can handle very large loads. The same company provides a different method of taking a full cross-section cut. The rotating-scoop sampler collects the sample from the entire stream at the discharge end of a conveyor belt (Figure 3-10). The scoop does not cut through the width of the stream as the cutter samplers do, but swings directly into the entire outfall head-on.

The Bristol Engineering Company has designed a rotary arm scoop sampler which takes a sample directly from a moving belt (Figure 3-11). The scoop is situated above the belt, then swings down, passing through the coal while moving in the same direction but slightly faster than the belt. At the top of its arc, the scoop is stopped and empties into a



| Model Travel (inches) | Feed Rate (TPH) | Feed Opening Size | Cutter Opening |
|---|---|---|---|
| 12-FI | 0-25 | 6" x 6" | 1" - 3" |
| 18-FI | 25-100 | 10" x 10" | 1" - 3" |
| 30-FI | 100-250 | 10" x 18" | 4" - 10" |
| 24-FI* | 0-25 | 10" x 10" | 1" - 3" |

Figure 3-5. Denver Equipment Division's chute sampler.

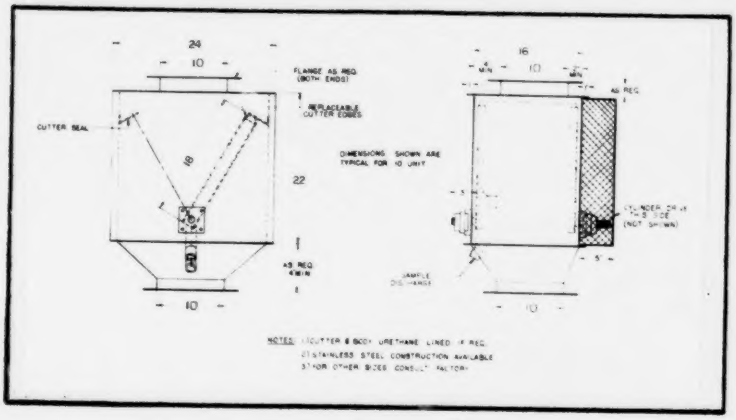

24
10
FLANGE AS REQ.
(BOTH ENDS)
REPLACEABLE
CUTTER EDGES
CUTTER SEAL
18
22
DIMENSIONS SHOWN ARE
TYPICAL FOR 10 UNIT
AS REQ.
4 MIN
10
16
4 MIN
10
2 MIN
AS REQ.
CYLINDER DRIVE
THIS SIDE
(NOT SHOWN)
5
SAMPLE
DISCHARGE
10
NOTES: 1) CUTTER & BODY URETHANE LINED IF REQ.
2) STAINLESS STEEL CONSTRUCTION AVAILABLE
3) FOR OTHER SIZES CONSULT FACTORY

Figure 3-6. Quality Control Equipment Company's cross cutting sampler.

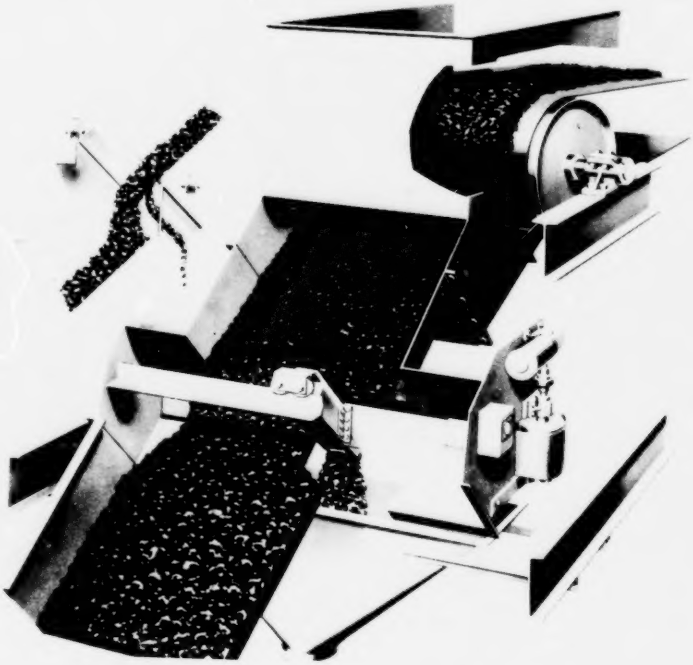


Figure 3-7. McNally belt sampler.

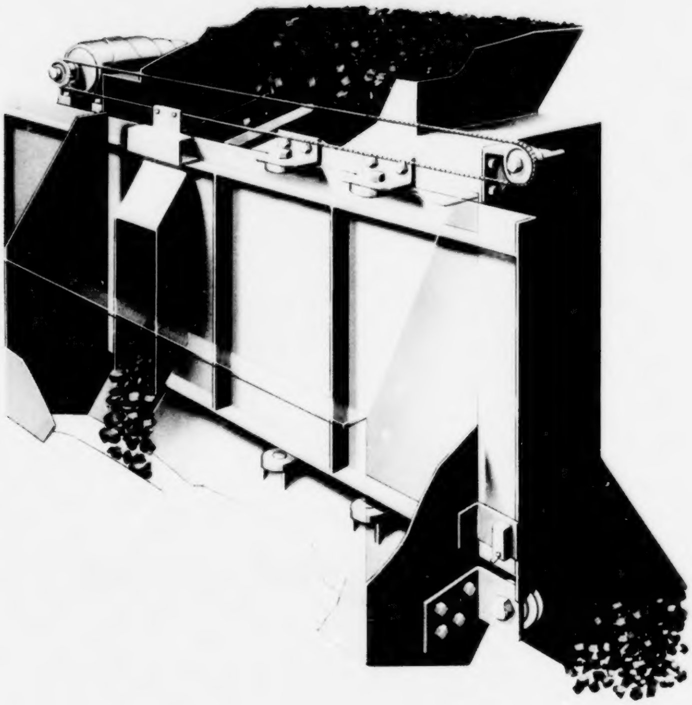


Figure 3-8. McNally plate sampler.

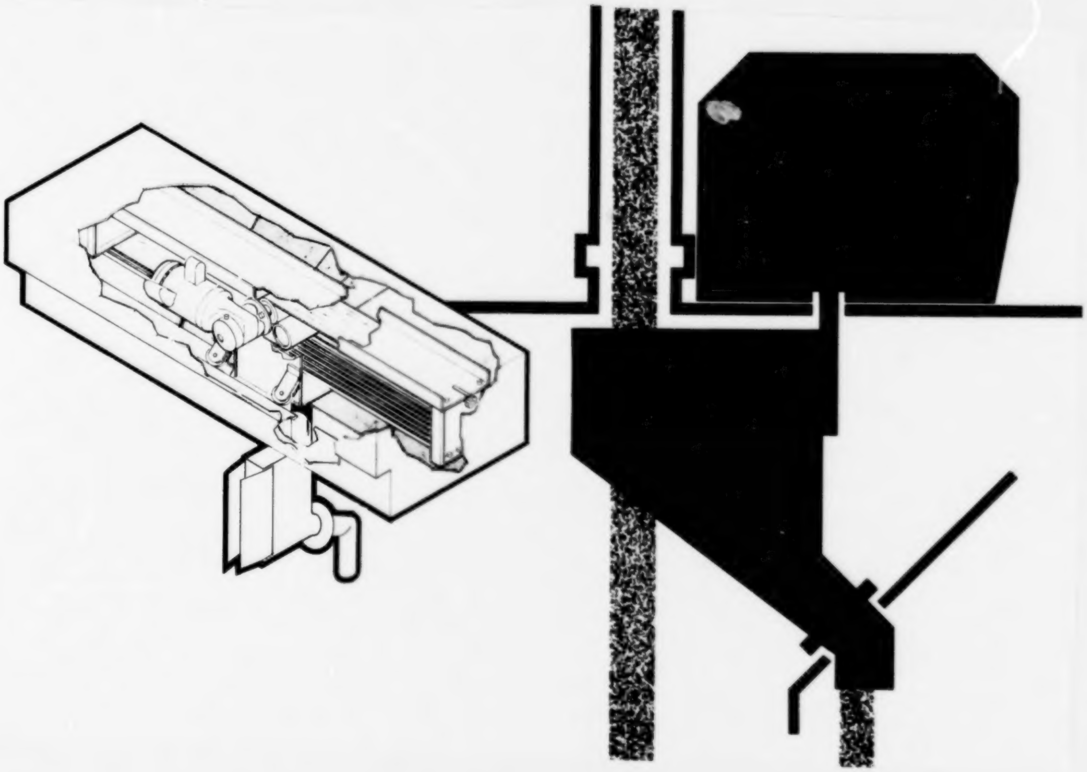


Figure 3-9. Harrison R. Cooper Systems, Inc. traversing cutter.

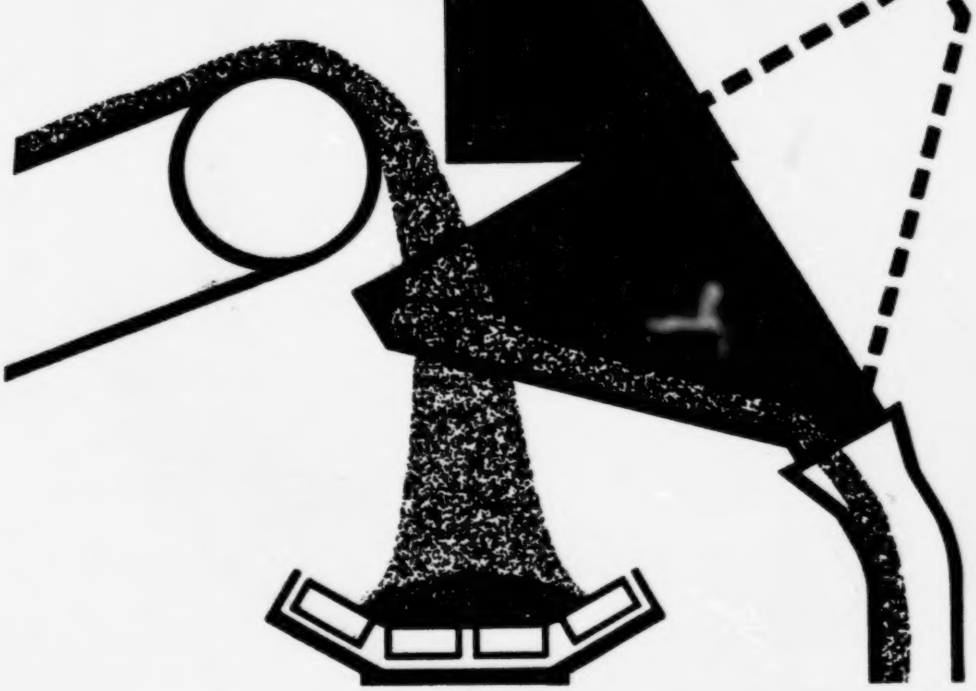


Figure 3-10. Harrison R. Cooper System's rotary scoop sampler.

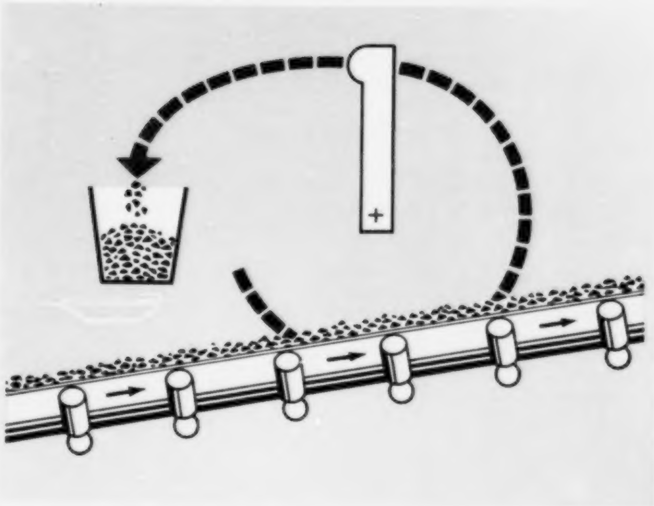


Figure 3-11. Bristol Engineering Company's rotary arm scoop.

hopper. This system takes only a 3-inch wide sample from the flow, a partial cross-stream cut. The validity of this sample is dependent on a homogeneous, non-stratified flow of material on the belt. The collection of a sample by the rotary arm scoop may also be subject to some segregation due to size, because it takes the sample from the same section of belt each time.

The many different approaches to mechanical sampling allow for many different applications and consideration of specific requirements. Although mechanical sampling is more precise than manual, not all mechanical methods collect equally representative samples. The design should include a means for taking a sample from the entire cross-section of the coal stream. Other important factors to consider are the speed of the cut and the size of the cutter opening.

In general, the cutter should move at a uniform speed to ensure that the entire cross-section is represented in equal proportions. The speed should also be slow enough to prevent segregation and rejection of particles due to disturbances of the coal stream. The cutter opening should be large enough, at least three times the size of the larger coal particles, to allow equal representation of all size particles.

The preferred method of sampling would be the method that obtained the most precise, representative increment. Table 2 shows the order of preference for each sampling scheme discussed in this section.

### 3.4 SAMPLING CONSIDERATIONS

It is useful when considering a sampling system to have as a goal a desired end result. The goal may be the collection of a truly representative sample of the coal entering a boiler, perhaps to assess the efficiency of the boiler or to aid in designing ash handling systems. Coal sampling is also used to determine a coal's characteristics, and subsequent market price. Coal sampling and analysis is now being used as a continuous $SO_2$ emissions monitoring method.

TABLE 2. ORDER OF PREFERENCE OF SAMPLING PROCEDURES AND METHODS

| Collection Procedure | Collection Method | Order of Preference |
|---|---|---|
| Stopped belt cut | Mechanical, automatic system | 1 |
| Stopped belt cut | Manual sampling | 2 |
| Full stream cut | Mechanical, automatic system | 3 |
| Full stream cut | Manual sampling | 4 |
| Part stream cut | Mechanical, automatic system | 5 |
| Part stream cut | Manual sampling | 6 |
| Stationary sampling | Mechanical, automatic system | 7 |
| Stationary sampling | Manual sampling | 8 |

In general, if the amount of sulfur present in the coal going into the boiler is known, then the $SO_2$ emission level can be determined by quantifying the amount of sulfur lost in combustion and trapped by air pollution control devices. With emissions monitoring as a goal, several factors must be considered when implementing a coal sampling plan. The first consideration is the accuracy of the sampling procedures. A choice must be made concerning manual versus automatic sampling and a stopped belt technique versus a moving full-stream cut. The number, frequency, and size of increments are also important considerations. Other important factors are the behavior of the coal in the boiler, and the amount of sulfur is removed by pollution control devices such as electrostatic precipitators and flue gas desulfurization systems.

The accuracy of sampling systems should be the most important characteristic considered when designing a sampling protocol. An automatic sampling system utilizing the stopped belt technique is the preferred system because it is the most precise. The number, frequency, and size of increments should be based on a statistical determination of the minimum requirements for a representative sample of that specific type of coal. The ASTM standard D 2234 is one such accepted statistical guide. The number, frequency, and size of increments is a very important factor to consider especially in cases of coal with high sulfur variability.

The accuracy of a sampling procedure can be determined by running a bias test. A bias test is a statistical comparison of samples obtained from the plant's routine sampling plan and samples obtained from a stopped belt technique. In the case of a mechanical sampling system, the sampler would be run for a set length of time and the final samples retained for analysis. Periodically during the run of the sampler, the belt would be stopped and a full cross section of the coal removed and retained for analysis. The length of the cut should be at least 20 times the top size of the coal. The stopped belt sample can be divided using a riffle or mechanical divider. Each of the two sets of samples should be analyzed for the parameter for which the variance calculations are to be made. In the case of $SO_2$ emissions monitoring, analysis for total sulfur content should be made. A statistical comparison of the results is then used to determine the level of variance between the normal sampling point and the bias test sampling point. If the bias is within acceptable limits, then the sampling system is considered free of bias.

Another important factor that must be considered when using coal sampling and analysis as an $SO_2$ emissions monitoring method is the correlation of the sulfur in the coal to the $SO_2$ emissions. First the time lapse between sampling of the coal and burn of the lot of coal that the sample represents must be determined. If the coal is sampled from a belt which feeds the coal storage bunkers, the retention times of the bunkers should be known. An 8-hour retention time would mean that the coal burn and resulting $SO_2$ emissions would correspond to the coal sampled 8 hours beforehand.

The amount of sulfur reduction resulting from the combustion in the boiler, and from air pollution control devices must be considered. The boiler efficiency, and efficiency of the sulfur removal processes such as flue gas desulfurization and electrostatic precipitation can be used to calculate the amount of sulfur going out the stack.

## SECTION 4

## ASSESSMENT AND STATISTICAL EVALUATION OF SAMPLE PREPARATION AND ANALYTICAL METHODS

In this section, the industry accepted procedures for preparing a coal sample for analysis and analytical methods currently in use are discussed. Sample preparation techniques such as air-drying, crushing, sample division, pulverizing, and mixing are presented, along with the equipment necessary to perform each step. Factors that create a sample bias or increase the variance are identified.

The analytical methods used to determine moisture, total sulfur, ash content, and calorific value of the samples are described, including the equipment needed to perform these analyses. Quality control measures to ensure the reliability of the analytical method and the precision of the data obtained are suggested.

### 4.1 SAMPLE PREPARATION

The collection of a representative gross sample using manual or automatic methods often yields a bulk quantity of material which may weigh as much as several hundred pounds. Normally, 50 to 100 grams of material are necessary to meet the analytical requirements of most coal characterization tests. The method used to reduce the gross sample to the analytical sample must maintain the integrity and representativeness of the sample while meeting the particle size and weight requirements as specified by the particular analytical methods.

The preparation of a gross sample for coal analysis requires several processing steps. Air drying is used to bring the moisture content of the sample to equilibrium with the air in the room where further preparation will take place. Samples are reduced or crushed from a nominal

3-inch top size to minus No. 4 or No. 8 mesh used to optimize the reproducibility of subsequent processing procedures. Sample division or splitting results in a smaller quantity of material without a loss in the sample's representativeness. Another sample reduction process, pulverizing to minus 60 mesh, is generally required for specific analytical procedures. Thoroughly mixing and homogenizing the analytical sample is necessary so that the small aliquots which are taken for individual tests are representative of the sample.

### 4.1.1 Air Drying

Air drying of a gross sample is generally the first step in sample preparation. The purpose of air drying coal is to bring its moisture content to equilibrium with the air in the room where it is to be reduced and divided. The gross sample can be air dried either in an air drying oven or on a drying floor. The temperature in an air drying oven should be 10°C to 15°C above room temperature with a maximum oven temperature of 40°C to prevent rapid oxidation of the coal. The coal is spread thinly on trays in the oven and exposed to a flow of slightly heated air (Figure 4-1). According to the ASTM D2013-72(1978) procedure(s), air drying should stop when the loss in weight of the gross sample is less than 0.1 percent/hour. Air drying on a drying floor is a much longer process, because there is no heat source such as an oven. The sample must meet the same drying time requirements as those described when using an air drying oven.

To minimize moisture variability between samples, several precautions are necessary. Air drying by oven must not be done for an excessive amount of time. Oxidation of the coal must be avoided. Air drying on a drying floor must be done in a room free from dust and excessive wind currents. These precautions will minimize the variance in this stage of sample preparation.

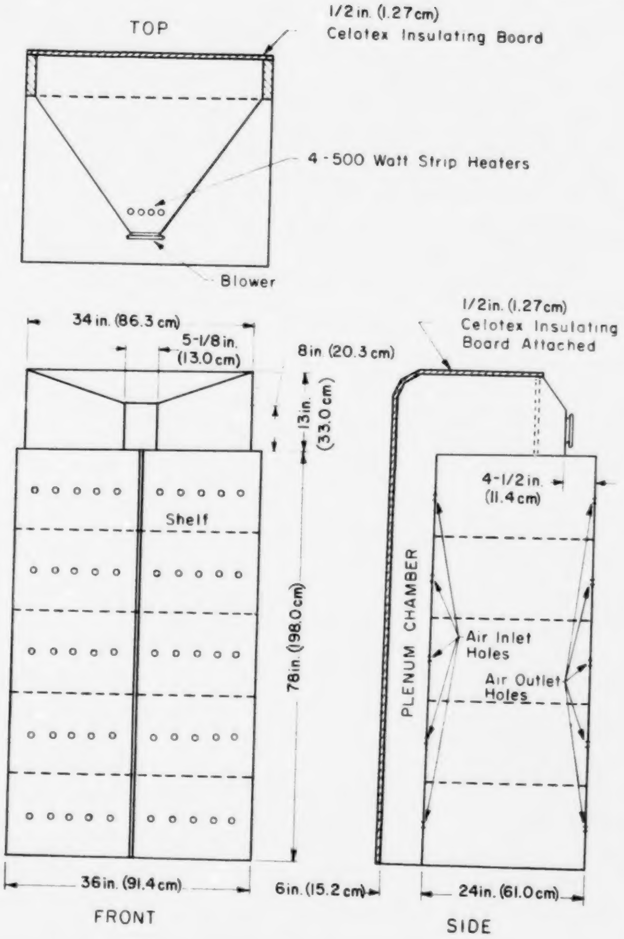

TOP
1/2 in. (1.27 cm)
Celotex Insulating Board
4-500 Watt Strip Heaters
Blower
34 in. (86.3 cm)
5-1/8 in. (13.0 cm)
8 in (20.3 cm)
13 in. (33.0 cm)
Shelf
78 in. (198.0 cm)
36 in. (91.4 cm)
FRONT
1/2 in. (1.27 cm)
Celotex Insulating Board Attached
4-1/2 in. (11.4 cm)
PLENUM CHAMBER
Air Inlet Holes
Air Outlet Holes
6 in. (15.2 cm)
24 in. (61.0 cm)
SIDE

Figure 4-1. **Air drying oven.**

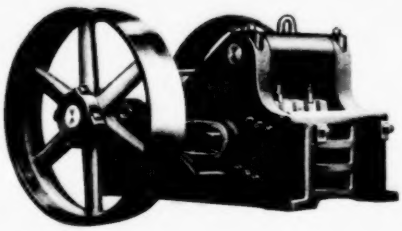
### 4.1.2 Sample Reduction

Sample reduction is the next step in sample preparation. Reduction is the process where a sample is reduced in particle size, without a change in weight. Crushers that are commonly used, include: jaw type (Figure 4-2), hammer mill (Figure 4-3), and rotary (Figure 4-4).

The crusher should be large enough to process the gross sample in a reasonable amount of time. A sieve test should be performed periodically on the product of the sample crusher, to assess the uniformity of particle size. Also, a uniform flow rate into the crusher should be maintained to guard against segregation by composition. This may introduce a bias if, during periods of a low flow rate to the crusher, the more brittle particles are selectively crushed first. A final precaution is to make certain that no particles remain in the crusher after this processing step is completed. The material must be crushed to a top size to pass through a No. 4 (4.75 mm) or No. 8 (2.36 mm) sieve based upon criteria established in ASTM D-2013-72(1978).

### 4.1.3 Sample Division

The next step in sample preparation is sample division. Here the sample is reduced in weight without a change in particle size. Riffles or sample splitters are used to divide the sample stream into two equal parts (Figure 4-5). One part is discarded, and the other remains as the sample for further splitting or analysis. It is important that the riffle divisions are at least three times the size of the coal being divided. The coal should be fed into the riffle in a slow, uniform stream covering all of the slots of the riffle. The coal should not be allowed to build up in or above the riffle slots, and it should flow freely through the slots. These precautions are necessary to minimize the variance due to riffle division.

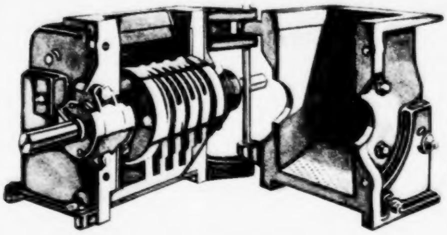


**Jaw Crushers**

| Size (") | Capacity Tons/Hr.* | Horsepower | Length | Width | Height | Weight (Lbs.) |
|---|---|---|---|---|---|---|
| 2 x 6 | ½ | 1 | 2'-10" | 2'-0" | 2'-0" | 1000 |
| 4 x 8 | 1 | 3 | 3'-10" | 3'-0" | 3'-0" | 3000 |
| 5 x 10 | 1-3 | 5 | 4'-10" | 3'-0" | 3'-4" | 4000 |
| 6 x 15 | 3-5 | 10 | 6'-0" | 4'-7" | 3'-9" | 8400 |
| 8 x 10 | 5-10 | 10 | 5'-0" | 3'-3" | 3'-4" | 4000 |
| 8 x 20 | 10-15 | 20 | 5'-8" | 5'-3" | 3'-5" | 9500 |
| 10 x 15 | 10-20 | 20 | 7'-6" | 5'-0" | 4'-4" | 12000 |

*Hard granite as feed

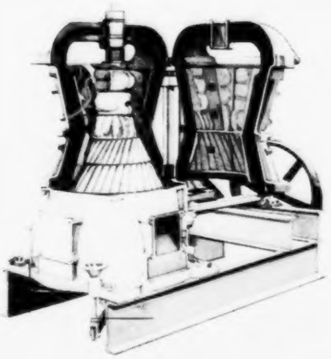
Figure 4-2. Jaw crusher.



***Hammermills***

| Size | Capacity Tons/Hr.* | Horsepower | Length | Width | Height | Weight (Lbs.) |
|---|---|---|---|---|---|---|
| 00SM | 1-1½ | 3-5 | 1'-1" | 2'-4" | 1'-5" | 500 |
| 00HP | 2-4 | 5-20 | 2'-5" | 3'-0" | 1'-11" | 1140 |
| 0SM | 2-9 | 10-30 | 4'-3" | 4'-1" | 3'-1" | 3700 |
| 09HP | 2-9 | 5-25 | 3'-7" | 4'-2" | 2'-9" | 2000 |
| 1SM | 6-20 | 25-75 | 5'-1" | 5'-8" | 3'-9" | 7000 |
| 018HP | 3-12 | 15-20 | 3'-7" | 4'-11" | 2'-9" | 3000 |
| 2SM | 12-70 | 50-100 | 6'-1" | 7'-0" | 4'-5" | 14000 |
| 024HP | 5-15 | 15-20 | 3'-7" | 5'-8" | 2'-9" | 3300 |

Figure 4-3. Hammermill crusher.

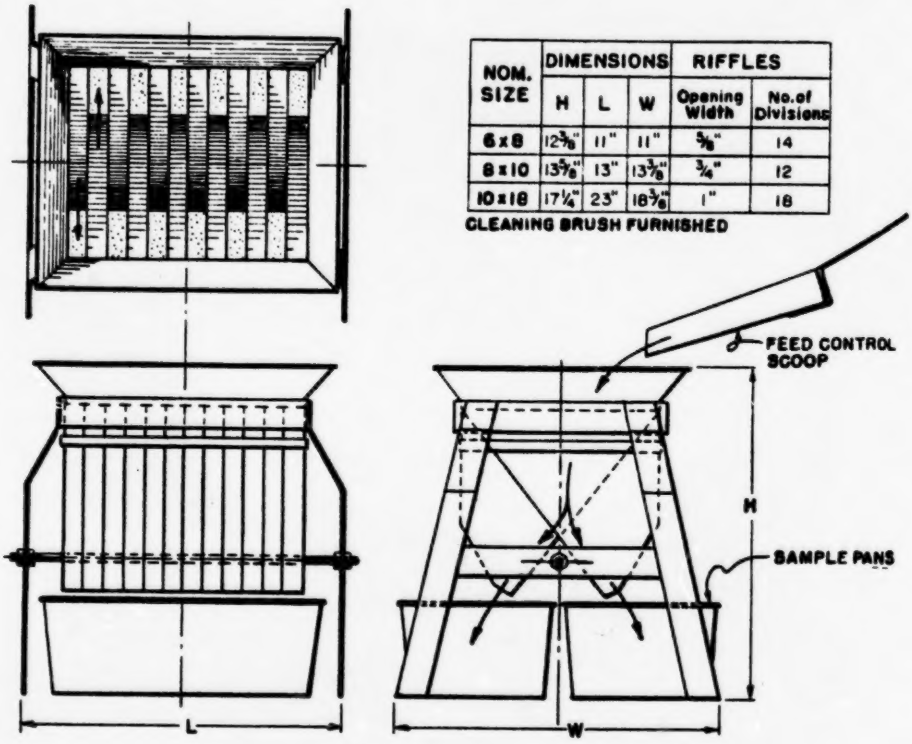


***Rotary Crushers***

| Size | Max. Feed Size Cubes | Capacity Tons/Hr.* | Horsepower | Length | Width | Height | Gross Weight (Lbs.) |
|---|---|---|---|---|---|---|---|
| 00 | 3" | 1 | 2 | 4'-5" | 2'-5" | 3'-5" | 1475 |
| 0 | 3" | 2 | 5 | 4'-7" | 3'-3" | 3'-10" | 1875 |
| 1 | 6" | 4 | 10 | 6'-4" | 3'-11" | 5'-0" | 4875 |
| 1½ | 8" | 6 | 15-20 | 7'-3" | 4'-4" | 6'-3" | 6800 |
| 2 | 8" | 10 | 20-25 | 8'-8" | 5'-5" | 7'-4" | 11000 |

*Limestone as feed

Figure 4-4. Rotary crusher.


FEED CONTROL SCOOP
H
SAMPLE PANS
L
W

| NOM. SIZE | DIMENSIONS |  |  | RIFFLES |  |
|---|---|---|---|---|---|
|  | H | L | W | Opening Width | No. of Divisions |
| 6 x 8 | 12 3/8" | 11" | 11" | 5/8" | 14 |
| 8 x 10 | 13 5/8" | 13" | 13 3/8" | 3/4" | 12 |
| 10 x 18 | 17 1/4" | 23" | 18 3/8" | 1" | 18 |

CLEANING BRUSH FURNISHED

Figure 4-5. Riffle.

Samples can also be reduced in bulk by mechanical sample dividers. Although not all automatic sample dividers split the sample in halves, accurate division can be achieved by extracting and compositing a large number of increments from the sample. The ASTM D2013-72(1979) procedure specifies that at least 60 increments be taken. One type of mechanical sample divider which does halve the sample is the rotary plate divider (Figure 4-6). The rotating plate directs the sample by centrifugal force toward the outlet.

Another type of mechanical divider is the rotating cutter (Figure 4-7). Ramsey/Galigher makes a rotary sampling machine with a sample cutter that revolves on a central shaft. The cutter opening and speed can be set so that a specific percentage of the sample is divided and remains as the sample for analysis.

The Denver Equipment Division's Vezin sampler is another rotary cutter sampler that can be used for sample division (Figure 4-8). The cutter turns at a constant speed and splits a fixed percentage of the sample.

A rotary sampler is also furnished by the McNally Pittsburgh Manufacturing Corporation. It is essentially a rotating drum with a cutter that takes a full stream cut once per revolution (Figure 4-9).

All of these rotary samplers are usually incorporated into a sampling system specified by the respective manufacturers. A complete sampling system generally includes the primary sampler, secondary sampler, crusher, and tertiary sampler. This is known as a three-stage sampling system (Figure 4-10). The primary increment is a full cross-stream cut from the total lot of coal. The secondary increment is a full cross-stream cut of the primary increment. After this stage, a crusher is used for sample reduction. Sample division is accomplished by a tertiary sampler which takes either a full cross-stream cut of the crushed secondary increment, or splits the secondary increment in half.

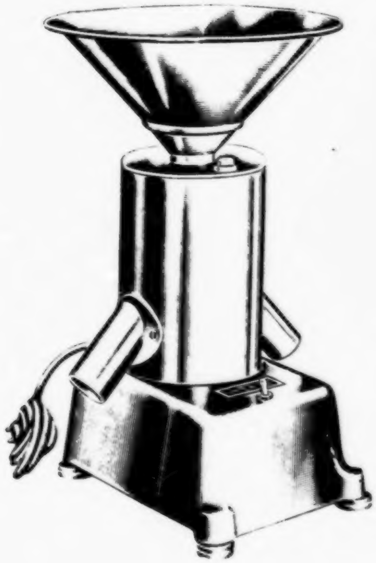
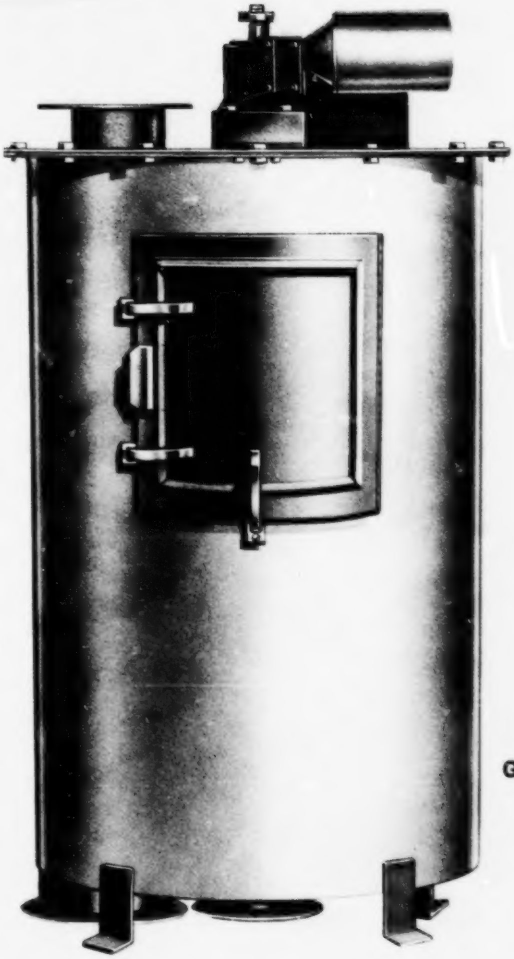


Figure 4-6. Rotary plate divider.



**GMD-500**

## CONDENSED SPECIFICATIONS

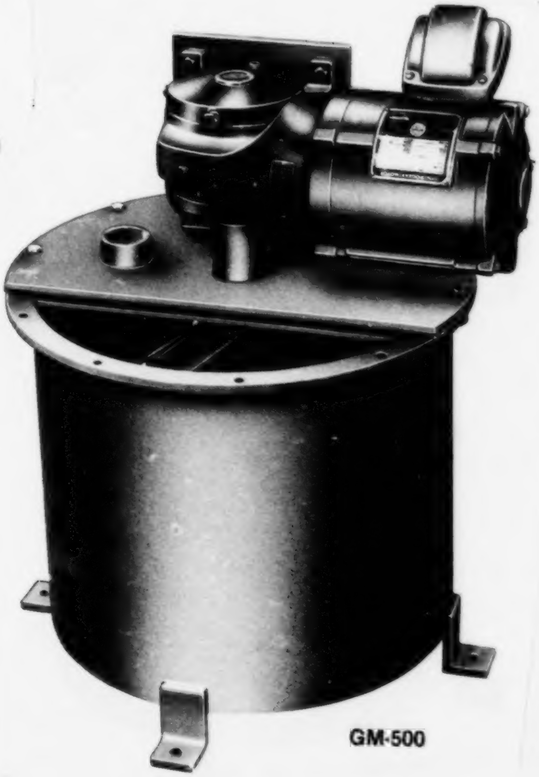
**GMD-500 Self-Contained Rotary Sampler**

| | |
|---|---|
| Drive: | electric |
| Speed:* | 18 in./sec. |
| Cutter Opening: | 0 = 2-1/4" |
| Infeed: | 6" diameter pipe |
| Capacity: | 25 tph<br>to 3000 GPM |

**GM-500 Self-Contained Rotary Sampler**

| | |
|---|---|
| Drive: | electric |
| Speed:* | 18 in./sec. |
| Cutter Opening: | 0 - 1-1/4" |
| Infeed: | 3" diameter pipe |
| Capacity: | 500 lbs. per hour |
| Particle Size: | -8 mesh |

**NOTE: at center of cutter*



**GM-500**

Figure 4-7. Ramsey sampler/Galigher tertiary rotary.

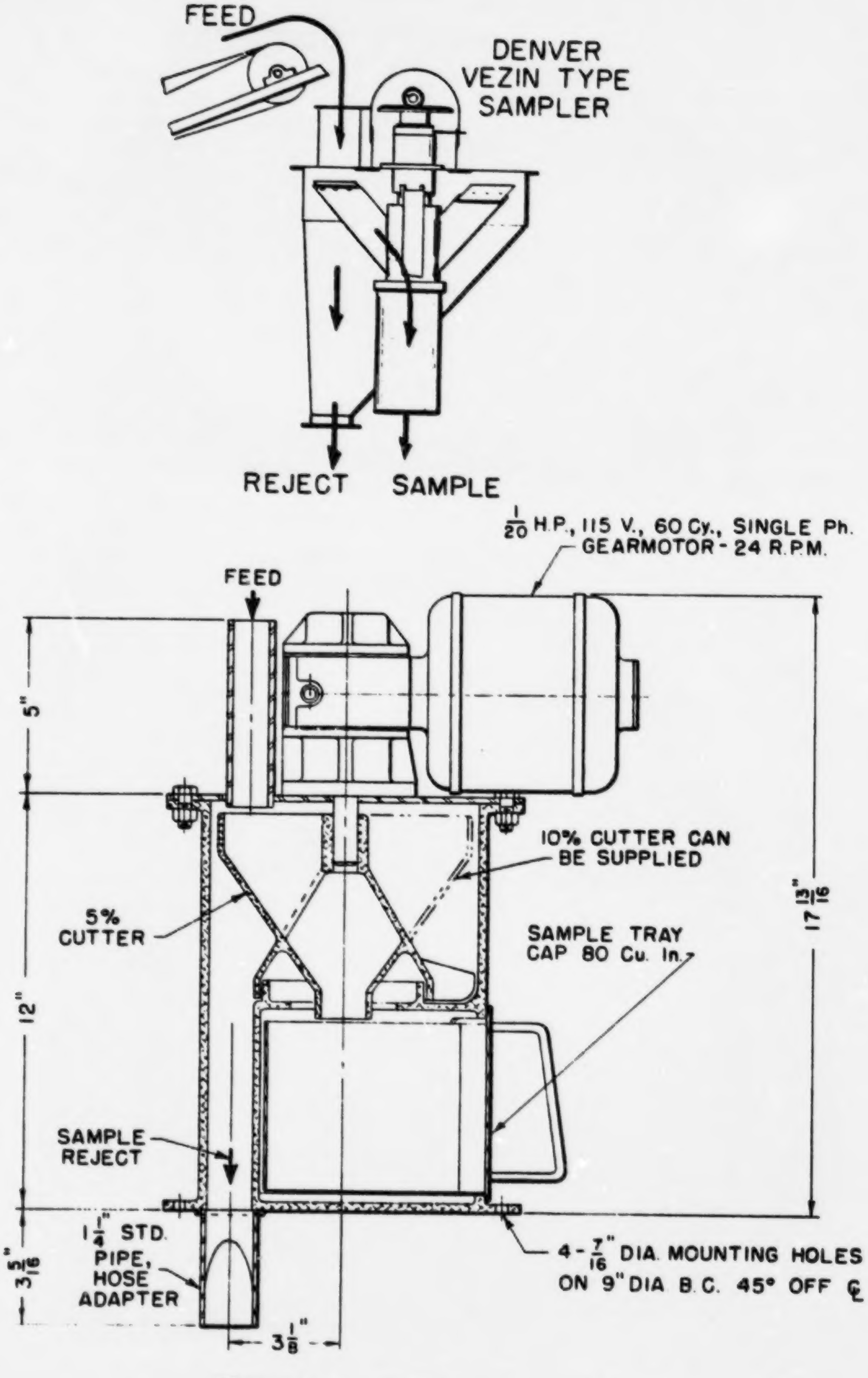

FEED
DENVER VEZIN TYPE SAMPLER
REJECT
SAMPLE
1/20 H.P., 115 V., 60 Cy., SINGLE Ph. GEARMOTOR - 24 R.P.M.
FEED
5"
12"
17 13/16"
10% CUTTER CAN BE SUPPLIED
5% CUTTER
SAMPLE TRAY CAP 80 Cu. In.
SAMPLE REJECT
1 1/4" STD. PIPE, HOSE ADAPTER
3 5/16"
3 1/8"
4 - 7/16" DIA. MOUNTING HOLES ON 9" DIA B.C. 45° OFF ℄

Figure 4-8. Denver Vezin sampler.

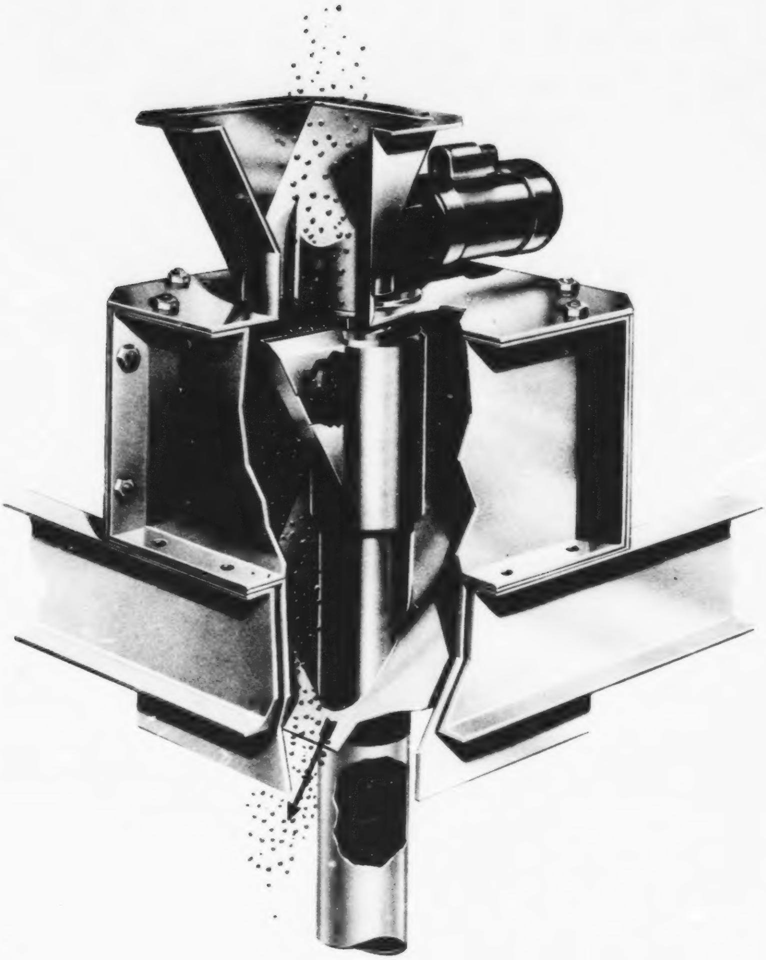


Figure 4-9. McNally rotary drum sampler.

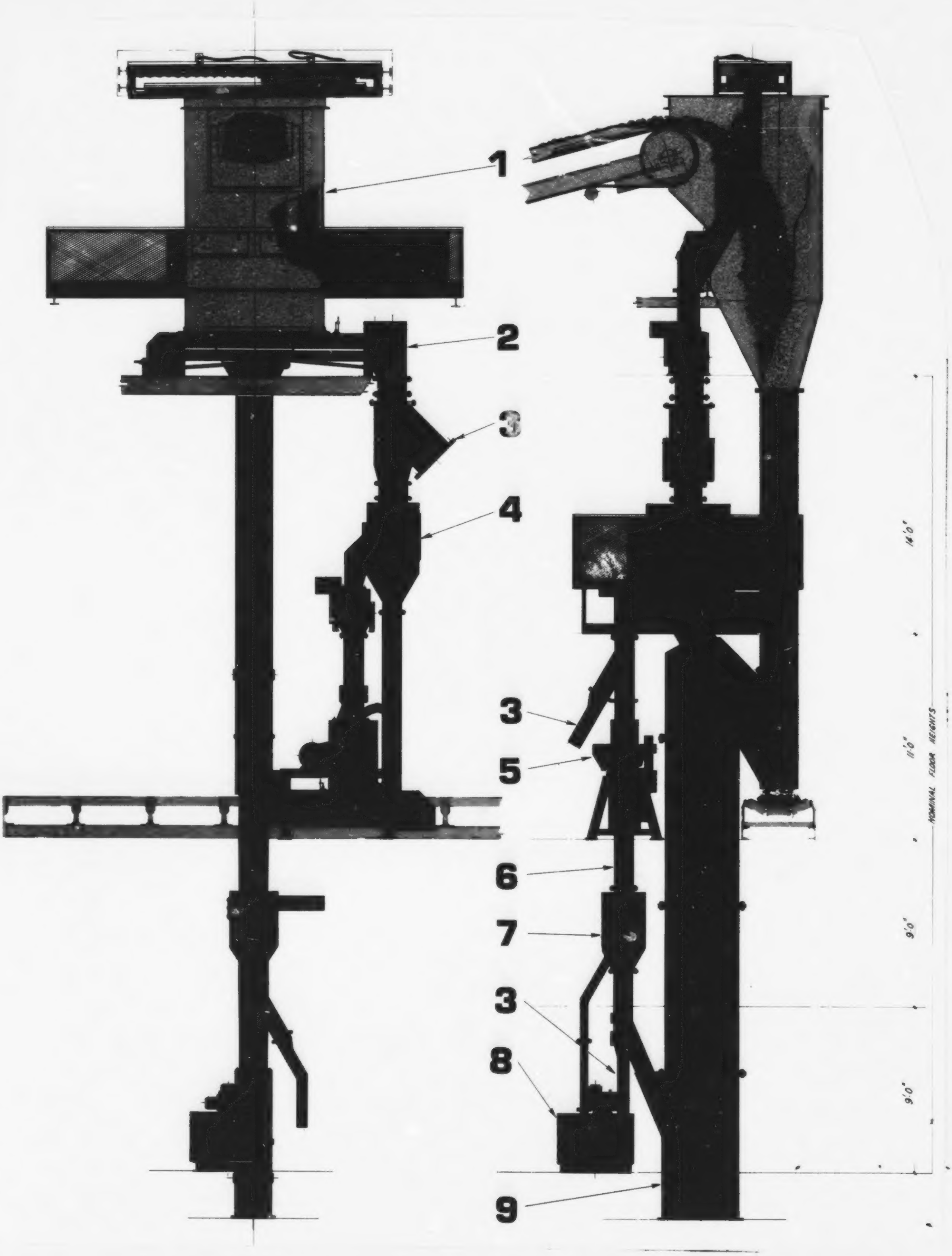

1
2
3
4
3
5
6
7
3
8
9
14'0"
11'0"
9'0"
9'0"
NOMINAL FLOOR HEIGHTS

Figure 4-10. Ramsey three stage sampling system

1 - Primary sampler
2 - Belt feeder
3 - Bias test chute
4 - Secondary sampler
5 - Crusher
6 - Sample chutes
7 - Tertiary sampler
8 - Sample collection
9 - Reject

Rotary plate samplers and rotary cutters are examples of tertiary samplers. Several sampling equipment manufacturers can supply a three-stage sampling system.

### 4.1.4 Sample Reduction for Analysis

For laboratory analysis, the sample must be further reduced to pass a No. 60 (250 um) sieve. A pulverizer is used to grind the sample. Suitable pulverizers include the Braun laboratory pulverizer (Figure 4.11) and the Sturtevant sample grinder (Fig. 2.12), which can produce a sample between 10 and 100 mesh.

Suitable precautions should be taken to ensure a continuous steady flow of coal into the pulverizer to ensure uniformity of sample particle size and composition. The ground sample is then divided by use of a small riffle (Fig. 4.13) until approximately 50 to 100 grams remain.

### 4.1.5 Sample Mixing

To ensure the homogeneity of the 50 to 100 gram analytical sample, a mechanical mixing device is employed. A mixing wheel (Fig. 4.14) can be used to rotate the sample containers so that the particles fall gently from the top to the bottom of the container. To obtain good mixing, sample containers should be between one-half and two-thirds full and the wheel should rotate at a slow speed. To be most effective, samples should be mixed just before portions of the sample are extracted for analysis.

## 4.2 SAMPLE ANALYSIS

Proximate and ultimate analyses are often used to characterize selected coal properties. Proximate analysis refers to the determination by prescribed methods of moisture, volatile matter, fixed carbon (by difference), and ash. Ultimate analysis includes the determination of carbon and hydrogen in the material (as found in the gaseous products

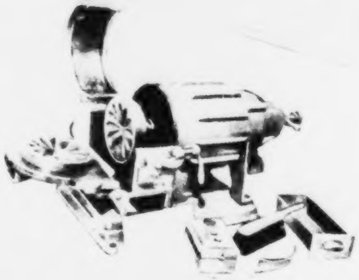


Figure 4-11. Braun lab pulverizer.

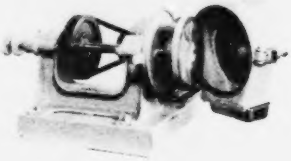


Figure 4-12. Sturtevant sample grinder.

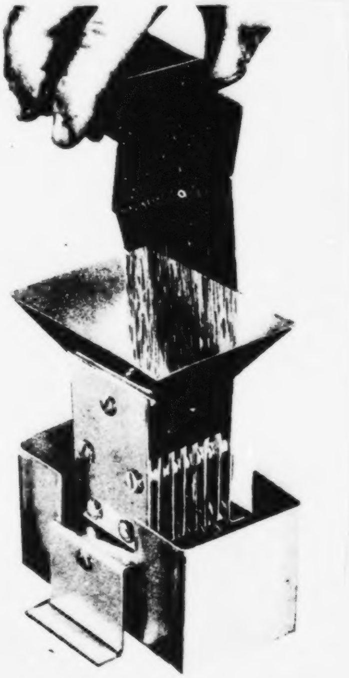


Figure 4-13. Small lab riffle.

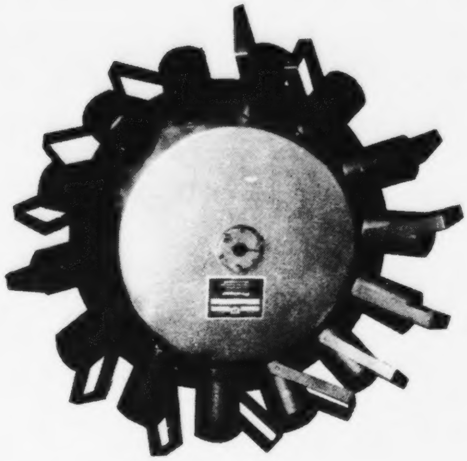


Figure 4-14. Mixing wheel.

of its complete combustion), sulfur, nitrogen, and ash (in the materials as a whole) and the estimation of oxygen by difference (ASTM D121-78). Although it is common to find several different methods for measuring each parameter, it is generally through agreement between parties, such as the coal supplier and customer, that specific methods and operating conditions are selected.

The following description provides details on the determination of moisture, ash, total sulfur, and gross calorific value (heat content) in coal.

### 4.2.1 Determination of Moisture

The determination of moisture in coal is calculated from the results of two processes: air drying and the determination of residual moisture. Air drying is a partial drying of the sample to equilibrate it with the room atmosphere in which sample reduction and division takes place. The total sample is weighed before being put into an air drying oven or being spread onto a drying floor. The sample is dried until the weight loss is not more than 0.1%/hour. The percent air - dry loss is calculated according to ASTM standard D3302 - 74(6) superior type as:

$$A = (L/G)100$$

where:

A = air - dry loss, %
L = weight loss due to air drying
G = weight of gross sample

Air drying can be done as described earlier in an air drying oven or on a drying floor.

The ASTM procedure for determining residual moisture requires the use of a forced-air oven under strict temperature, time, and air flow specifications. A one gram sample of coal is heated in a forced air oven (Fig. 4-15) at between 104°C and 110°C for exactly one hour. The oven must be capable of renewing the heated air two to four times each minute. The air current must be dried by passing through sulfuric acid or another suitable dessicant. The percent residual moisture is calculated as follows:

$$M = [(A - B)/A] \times 100$$

where:

$M$ = % moisture

$A$ = initial sample weight

$B$ = sample weight after heating

Quality control measures to ensure the reliability of moisture content data begin with strict adherence to the standard method. This lessens the consideration of the method as a variable. The reproducibility of results obtained by the same method, on splits of the same sample, performed by different laboratories should not differ by more than 0.50%, according to the ASTM(6). A duplicate sample check should not differ by more than 0.30%. Duplicate determinations should be carried out frequently. Other routine control checks should include periodic verification of temperature settings and preventive maintenance of equipment.

Another method for moisture determination in coal is by the direct gravimetric method. In this procedure, the analysis sample is heated in a retort tube to 105°C-110°C for one hour. A stream of pre-heated, oxygen free, dry nitrogen is passed through the retort which drives off the moisture. A weighing tube containing magnesium perchlorate, or another suitable dessicant, collects the moisture. The weight increase of the dessicant tube is the moisture content of the sample. The direct gravimetric procedure for determining moisture content in coal is a method recognized by the International Organization for Standardization (ISO) as standard method ISO 331.(18)

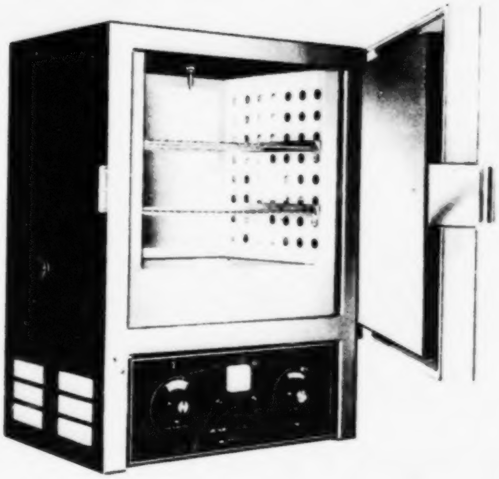


Figure 4-15. Forced air oven.

ISO requirements for quality control in terms of precision are a difference of less than 0.20% for reproducibility. Exact procedures, instrument calibration checks, and preventive maintenance all contribute to increased reliability and should be incorporated into a quality control plan.

Another moisture determination method is ISO standard 348(18), a direct volumetric procedure where the coal is distilled with toluene, carrying the moisture to a graduated receiver. The moisture condenses and separates from the toluene, allowing direct calculation of the volume of water released from the coal. An advantage of this method is that the sample is protected from oxidation. A sample of 50 to 100 grams is needed.

Quality control procedures for this method include routine analysis of duplicates which should not differ by more than 0.5%. A reproducibility check is not an accurate indicator of reliability since differing humidity levels in different labs would result in widely varied data.

Although the above methods are the standards for moisture determination, there are other accepted procedures. Automatic coal analyzers are being used for moisture determination. The speed of these systems is a great advantage. The Fisher Model 490 Coal Analyzer performs moisture determinations in an oven with controls to pre-set oven temperature and drying time. A microprocessor calculates percent moisture. The LECO Corporation also has an automatically programmable furnace suitable for moisture determinations. The procedure is similar to ASTM methods. Duplicate determinations should also be run for the automatic furnaces techniques to determine repeating and reproducing reliability.

### 4.2.2 Ash Determination

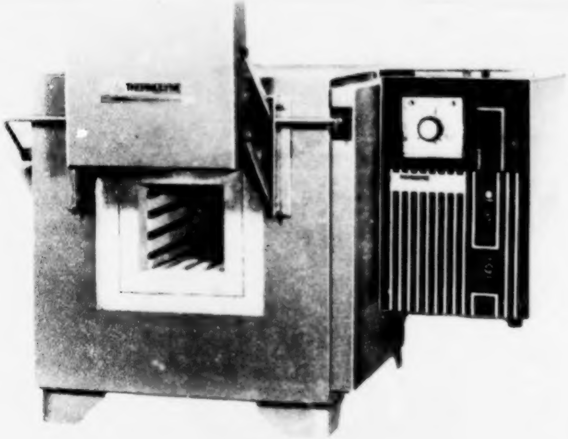
The determination of ash in a sample of coal is a weight loss procedure. The sample undergoes complete combustion, and the percent ash is calculated by weighing the residue. The ASTM standard D3174-73(6) recommends two approaches using either the dried sample from the moisture determination or a new one-gram sample.

When using the dried sample, a muffle Furnace (Fig. 4.16) is used to gradually heat the sample to 500°C in one hour and 750°C in two hours. The sample is then heated to constant weight (± 0.001 gram) at 750°C. The ash content is calculated as a percent weight loss.

When a new undried sample is used, it should be gradually heated in a cold muffle furnace until it glows red. The heating rate should be slow enough to avoid the loss of volatile matter. The combustion should be completed at 700°C to 750°C. Before being weighed, the sample must be allowed to cool in a desiccator.

Quality control measures to ensure the reliability of ash determination data are dependent on exact duplication of the procedures. To ensure complete oxidation of the pyritic sulfur and to remove the $SO_2$, a large supply of air must be available in the muffle furnace. The furnace should be capable of heating the sample slowly and of having its temperature adjusted between 700°C and 750°C. For samples with no carbonates present, the ASTM repeatability limit is 0.2%. It is 0.3% for samples with carbonate. Coals containing over 12% ash have a repeatability limit of 0.5%. The ASTM reproducibility limits are 0.3% for no carbonates present, 0.5% for coal with carbonates, and 1.0% for coals with more than 12% ash.

The ISO 1171(18) approach is similar but allows a faster heating rate and a higher maximum temperature. A one- to two-gram sample is heated to 500°C in 30 minutes and to 500°C to 815°C in 30 to 60 minutes. The final temperature reached at 60 minutes is maintained for an additional 60 minutes. The sample is then allowed to cool, weighed, and



| Operating temperature | Chamber size, W x H x D" |
|---|---|
| 2000 F/1093 C | 4 x 3¾ x 9 |
| 2000 F/1093 C | 4 x 3¾ x 9 |
| 2150 F/1177 C | 4 x 3¾ x 9 |
| 2150 F/1177 C | 4 x 3¾ x 9 |

Figure 4-16. Muffle furnace.

returned to the 815°C muffle furnace for 15 minutes or until the sample has attained a constant weight (± 1.0 mg). The ash percentage is determined from the weight loss.

The precision levels set by the ISO as a control are 0.2% for repeatability and 0.3% for reproducibility. The same precautions concerning procedural accuracy are necessary to ensure data reliability for this method.

More rapid methods are available for calculating ash content in coal samples. The Fisher Coal Analyzer and LECO Automatic Furnace use the standard furnace method, but the heating rate, transition temperature, final temperature, and duration of burn can be programmed into a microprocessor. The results are displayed as percent ash automatically. Another automatic method is X-ray fluorescence. The X-ray tube must be calibrated with a known standard, and the sample is compared against the standard. This method has not proved to be as accurate as the standard methods but is much quicker(19).

### 4.2.3 Determination of Total Sulfur

There are three standard methods for determination of total sulfur in the analysis sample of coal: the Eschka method, the bomb washing method, and the high-temperature combustion method(6).

The Eschka method requires 1.0 gram of sample pulverized to pass No. 60 sieve. The coal is mixed with 3.0 grams of Eschka mixture (2 parts MgO, 1 part $Na_2(CO)_3$). The mixture is placed in a porcelain or platinum crucible and covered with one gram of Eschka mixture to prevent sulfur lost as $SO_2$ during ignition. The crucible can then be heated slowly over a low flame for 30 minutes (gradually increasing the temperature until all black particles are combusted) or heated in a cold muffle furnace that reaches 800°C ± 25°C within one hour and maintained at 800°C ± 25°C until black particles are present.

After combustion, the soluble sulfur compounds are leached out and oxidized with bromine. The sulfur is then precipitated as barium sulfate ($BaSO_4$). The precipitate is filtered and then heated to 925°C until the sample is at constant weight. The ASTM requires a correction to be applied as part of this procedure because of the possibility of solubility error for $BaSO_4$. A standard sulfate should be analyzed in the same manner as the analysis sample. The correction factor is the difference in weight of $BaSO_4$ precipitated from the standard and the actual value for that standard.

The Eschka method is also recognized by the ISO 334(18). The recommended procedure is the same as the ASTM method. The ISO requires a blank determination be conducted simultaneously with the analysis sample to quantify any sulfur contamination from the atmosphere. A known amount of potassium sulfate is added to ensure a precipitate. The amount of sulfur detected in the blank, minus the known amount added, is considered as atmospheric contamination. This value is deducted from the result of the analysis sample. In addition, the ISO specifies the use of hydrogen peroxide to ensure complete oxidation of the sulfur to sulfate as opposed to the ASTM requirement of bromine water.

The percent sulfur content is calculated as:

$$S = \frac{(A - B) \times 13.738}{C}$$

where:

S = % sulfur

A = weight of $BaSO_4$ precipitated (grams)

B = weight of $BaSO_4$ correction (grams)

C = weight of sample used (grams)

Besides the quality control methods of repeatability and reproducibility, a correction procedure must be run on a standard sulfate as part of the control. The correction procedure must be an exact duplication of the sulfur determination procedure. A reagent blank determination can be run to quantify the amount of sulfur contamination from the atmosphere. The precision limits for repeatability are 0.05% for coals containing less than 2% sulfur, and 0.10% for higher sulfur coals. The reproducibility limits are 0.10% for less than 2% sulfur coal and 0.20% for higher sulfur coals.

The ASTM's bomb washing method uses the oxygen-bomb calorimeter washings from the determination of calorific value to calculate total sulfur content(6). The interior of the oxygen bomb (Fig. 4.17) is washed with a spray of water containing methyl orange until no acid reaction is observed. The washings are collected, and an acid correction titration is performed for calorific value determination. To precipitate the sulfur, $NH_4OH$ is used to reduce the pH to 5.5 to 7.0. The washings are then boiled and filtered. This residue is washed with hot water several times, and then 1 ml of bromine water is added and enough HCl to make the solution slightly acidic. The remaining steps of adjusting the acidity, precipitating the $BaSO_4$, and determining total sulfur content are the same as in the Eschka method.

The precision limits for repeatability and reproducibility are the same as the limits given by the ASTM for the Eschka method. Additional quality control techniques should be used such as a reagent blank determination and a standard sulfate determination.

Another sulfur determination recommended by the ASTM is the high-temperature combustion method(6). A 0.5 gram sample is burned in a tube furnace (Fig. 4.18) under a stream of oxygen to prevent formation of nitrogen oxides. The oven temperature is 1350°C, with the sample moving progressively closer to the hot zone to ensure a slow heating rate. After ten minutes, the sample is in the center of the hot zone and remains there for four minutes. Sulfur and chlorine oxides produced are collected in two absorption bottles containing hydrogen peroxide producing

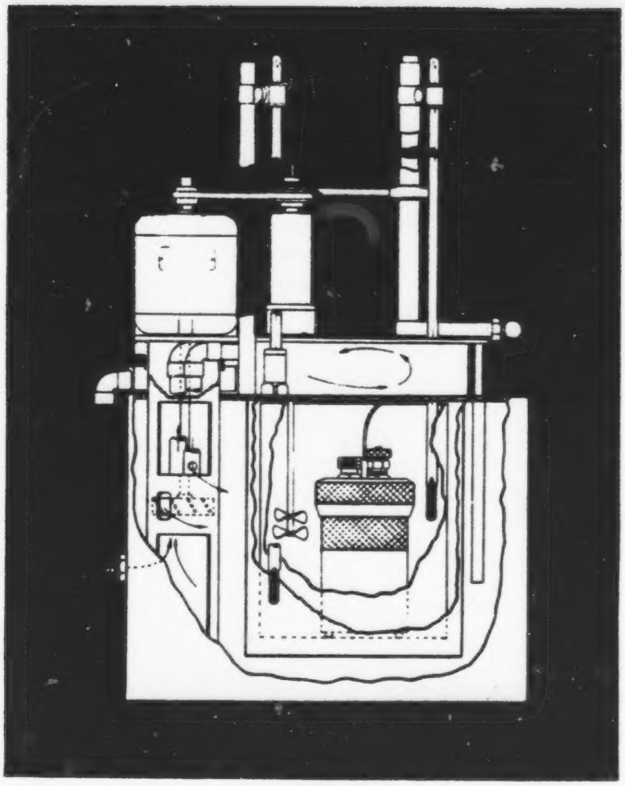


Figure 4-17. Adiabatic bomb calorimeter.

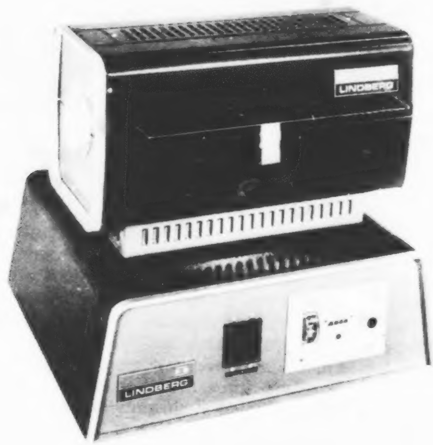

LINDBERG
LINDBERG

Figure 4-18. Tube furnace.

hydrochloric and sulfuric acids. The total sulfur is calculated from the total acidity which is determined by titration with standard NaOH and then with standard $H_2SO_4$.

The same procedures and equipment are described in the ISO standard 351 for determination of total sulfur by the high temperature combustion method(18). A furnace temperature of 1250°C and 1350°C can be used. The higher temperature allows a lower oxygen flow rate without the formation of nitrogen oxides. At the higher temperature, the sample should be covered with 0.5 grams of aluminum oxide. At 1250°C, 0.5 grams of kaolin or 0.15 grams of iron phosphate are used.

Strict adherence to the standard procedures, including standard reagents, is important for the control of this method. Results of duplicate determinations performed by the same lab should not differ by more than 0.05%. Reproducibility experiments performed or duplicates by different laboratories should not differ by more than 0.15% for coals containing less than 2% sulfur, and by more than 0.25% for higher sulfur coals.

There are many methods, besides the accepted standard methods, used by industry. Automatic analyses are widely preferred because of the short analysis time, but they are not well recognized for the reliability of their data.

A semi-automated procedure which the LECO Corporation introduced many years ago used a high temperature induction furnace to combust the coal and convert the sulfur to $SO_2$ in an oxygen rich atmosphere. The evolved $SO_2$ was automatically directed to a glass chamber where it was iodometrically titrated with standard sodium thiosulfate. This method has been replaced by a new LECO technique which uses a microprocessor to initially weigh the sample, control the combustion process, and calculate final sulfur concentration. This technique is covered by ASTM D-4239-83, "Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Method." This method is similar to the ASTM

D-3177-75 Method C high temperature combustion procedure. In the new high temperature tube furnace method, the sample is placed in a ceramic boat, weighed, and inserted into a resistance wire heated furnace. The evolved $SO_2$ is measured spectroscopically by an infrared detector and compared to calibration standards automatically. Total analysis take two minutes.

Another automatic analyzer based on the high temperature combustion method is the Preiser/MINECO sulfur analyzer. Although not as fast as the spectroscopic method, it offers excellent precision when compared with the Eschka method.

Another accurate and rapid system is the Fisher Sulfur Analyzer System. The sample is burned in a combustion-tube furnace. The oxidized sulfur compounds are absorbed by a diluent, resulting in a decrease in current in a preset platinum electrode. Titrant is automatically added to restore the current to the preset level. The amount of titrant needed is directly related to sulfur content. Analysis time is approximately two minutes.

Another combustion tube type of analyzer has a different sulfur detection scheme. The Perkin-Elmer Corporation's thermal-conductivity analyzer determines sulfur concentration by reading the differential response across two thermal conductivity cells. Accuracy is reported to be 0.3% of total sulfur.

Several companies make X-ray fluorescence analyzers capable of measuring total sulfur content in coal. The operating principle is generally the same. Low level radiation is emitted from an iron excitation source, causing X-rays that are characteristic of sulfur. These X-rays are measured and are proportional to the sulfur concentration.

To ensure reliability of the data obtained from rapid sulfur analyzing systems, the manufacturer's recommendations for equipment maintenance, standardization and calibration, and operating procedures should be closely followed. In addition, results of duplicate analyses should be within accepted repeatability and reproducibility limits for the determination of sulfur.

### 4.2.4 Determination of Calorific Value

The calorific value of coal is determined in a bomb calorimeter. The static isothermal and adiabatic methods are two accepted standard procedures. In the static isothermal method, a water jacket is used to protect the calorimeter from the effects of external heat. In the adiabatic method, the jacket temperature is continually adjusted throughout the procedure to match the temperature of the calorimeter, thereby minimizing heat exchange between the bomb and the surroundings. Because of this lack of heat transfer, no correction factor is needed in the adiabatic method. In both cases the gross calorific value is determined. The gross calorific value is the amount of heat produced by complete combustion of a specified quantity of coal under strictly specified conditions in an oxygen bomb calorimeter. The net calorific value is a lower value and can be calculated from gross calorific value. A suitable oxygen bomb calorimeter is shown as Fig. 4.17.

The procedure for the static isothermal method of determining gross calorific value includes a standardization procedure for calibrating the calorimeter. Ten separate calorific value determinations are run using benzoic acid pellets as the standard fuel source. The pellet weight should result in the same temperature rise as is expected with the coal to be tested. This allows the calorimeter's energy equivalent to be calculated. The bomb should be charged to the same oxygen pressure for each calibration and each calorific valve determination.

The calorific value is determined by burning a one-gram sample and observing the temperature rise of the calorimeter. The corrected temperature rise is calculated by making radiation corrections for heat contributed by other sources. The gross calorific value (Btu/lb) is then obtained by multiplying the corrected temperature rise by the energy equivalent which is then adjusted to account for energy formation from side reactions. The ASTM and ISO recognize the static isothermal method in their standards ASTM D 3286-77 and ISO 1928$^{(6,18)}$.

The adiabatic bomb calorimeter method is a similar process except that it requires control of the water jacket temperature. A standardization procedure is used to obtain the water equivalent of the calorimeter using benzoic acid as the standard fuel source. The pellet weight should result in the same temperature rise as would be expected for the analysis sample. The procedure should be exactly the same as for the calorific value determination.

A 1.0-gram sample (± 0.1 mg) is placed in the crucible, and 1.0 ml of water is added to the bomb. A measured length of firing wire is connected to the ignition terminals. The bomb is charged to an oxygen pressure between 20 and 30 atm. The pressure must be consistent for each calorific determination. During combustion, the jacket temperature must be continually adjusted to equal that of the oxygen bomb. The gross calorific value is calculated as the corrected temperature rise multiplied by the water equivalent, minus corrections for heat added by other sources, divided by the sample weight. ASTM standard D2015-77$^{(6)}$ and ISO standard 1928$^{(18)}$ are accepted procedures for determining gross calorific value by the adiabatic bomb calorimeter.

Quality control measures to ensure the reliability of data from the calorific value determinations include frequent calibration of the calorimeter, maintenance checks of equipment, and exact procedures. The repeatability checks should not differ by more than 50 Btu/lb. The reproducibility checks should not differ by more than 100 Btu/lb.

Other methods besides the bomb calorimeters are available for measuring calorific value. Several X-ray fluorescence systems have been developed which measure a radiation level which is proportional to Btu. The rapid analytical results available from these systems make them more advantageous, but calorific value determination by bomb calorimeter is a more proven method.

Automatic, microprocessor controlled, bomb calorimeters are available from the LECO Corporation and the Fisher Scientific Company. The entire process is preprogrammed and totally automatic. Both procedures are based on ASTM D 2015, the adiabatic bomb calorimeter method.

Manufacturers' specifications and instructions should be closely followed for all automatic systems. Calibration checks should be performed on a regular basis as should duplicate sample runs. The precision values resulting from repeatability and reproducibility tests should be within specified limits for the adiabatic bomb calorimeter method.

The heating value of coal may be computed using empirical relationships and the concentrations of various constituents(20). The Dulong-Petit formula is equal to:

$$\text{Btu/lb} = 145.44\ C + 620.28\ (H - O/8) + 40.50\ S$$

Where: C = carbon (% by weight - as received)
H = hydrogen (% by weight - as received)
O = oxygen (% by weight - as received)
S = sulfur (% by weight - as received)

Other relationships have been developed which may improve the accuracy of predicting the as-received heating values for certain types of coal. These include the Grummel-Davies formula and the T.K. Subramanian formula.

# SECTION 5

# SULFUR LOSS IN COAL-FIRED UTILITY BOILERS

Coal feedstock is usually sampled as it is bunkered. When it is removed, it moves on to the pulverizers and thence to the boilers. Sulfur is removed during pulverizing and combustion from pathways other than the emission of gaseous sulfur dioxide in the flue gas. Hence the amount of gaseous sulfur dioxide in the flue gas is not simply related to the measured sulfur in a CSA program. As sulfur dioxide is regulated by EPA, guidelines need to be developed to estimate the sulfur dioxide based on as-bunkered coal sulfur measurements. Several factors must be considered in this development: the coal characteristics (ash constituents, organic and pyritic sulfur content, and heating value), the combustion process (boiler size, firing type, and generating load), and possible fuel additives.

Information on sulfur loss has been collected by different utilities for specific boiler units; however most of this information has not been published. Other reports that address this subject on a more comprehensive basis are limited in scope. Therefore, the verbal comments of some of the utility sources contacted for this project have been used as the basis for this section.

Mass balance calculations for sulfur are extremely difficult to perform. One difficulty is that some of the sulfur is released as volatile compounds (which are inherently difficult to sample). A second is the difficulty in achieving truly representative samples of the coal entering the bunker, the pulverized coal entering the boiler, bottom ash leaving the boiler, fly ash collected by the emission control devices, and fly ash emitted to the atmosphere. A third is the difficulty associating a fixed coal lot sampled as-bunkered to the as-fired samples since the time between these samples is a random variable depending on the plant's bunker retention volume as well as the coal handling and storage system. A final problem is the difficulty in accurately measuring the mass of material entering and leaving the boiler (combustion air, coal, bottom ash, fly ash, and flue gas).

## 5.1 SULFUR LOSS IN PULVERIZERS

Coal contains sulfur in three forms: pyritic sulfur, contained in discrete particles or bands of sulfide minerals; organic sulfur, bound in the chemical structure of the coal; and sulfate sulfur, an oxidation product(21). Sulfate sulfur, usually as calcium or iron sulfate, is present only in concentrations below 0.1 percent(22). Organic and pyritic sulfur are both capable of being oxydized to gaseous sulfur oxides during combustion. The organic sulfur cannot be removed by mechanical means whereas some of the pyritic sulfur can be removed by the pulverizers.

The pulverizers crush the coal prior to firing. They have the capability of rejecting particles that are denser than coal. This material, known as mill rejects, typically consists of rock fragments, larger chunks of pyrite, and tramp metal. Eastern bituminous coals have high pyritic sulfur content in contrast to sub-bituminous and lignitic coals from the west(22). Thus, eastern coals contain sulfur in a form able to be rejected by the pulverizers. A mass-balance study(23) of a plant using western Kentucky coal showed a reduction of 0.3 percent; several utility representatives thought this figure typical, with maximal reduction 1 percent.

## 5.2 SULFUR LOSS IN COMBUSTION

During coal combustion, a small amount (typically 0.5 to 1.5 percent) of fuel sulfur is converted to $SO_3$, although the high flame temperatures and long residence times of modern utility boilers favor the formation of $SO_2$.(24) The proportion of $SO_2$ to $SO_3$ is controlled by the combustion area temperature, the amount of excess air supplied to the flame, and the presence of certain catalysts(21).

The most significant sulfur loss during coal combustion comes from reaction with coal-ash constituents and/or fuel additives (e.g., limestone) to form sulfates. Estimates range from 5 to 40 percent of the total sulfur would be lost(25) in the process.

## 5.3 GUIDELINES

In general, not enough investigation has been done in considering the effects and interrelationships of the various factors involved in sulfur retention to allow a blanket method to be suggested. However, some possibilities can be given.

a. A constant 5 percent rate seems a reasonable compromise between regulatory demands for conservancy and industrial demands for "reality".

b. Each utility can take a constant rate (say 5 percent) or can, using mass balance, prove it is entitled to more. The regulatory difficulty here is that the retention rate is so variable that past information may bear little relation to the future.

c. Allow no retention rate. This is the most conservative approach forcing the sources to bear the entire burden of the retention if they wish to use CSA.

d. Allow no retention in general but allow each source, using mass balance, to prove it is entitled to one. The drawback stated in (b) applies.

# SECTION 6

# COMMON COAL INDUSTRY AND UTILITY PRACTICES FOR COAL SAMPLING AND ANALYSIS

Several common coal sampling and analytical techniques are currently used by coal-fired utilities to evaluate their fuel. The purpose of this section is to determine the extent of use of these methods and any quality-control measures associated with them. This information is important to the study because it indicates how closely current industry practices match the proposed EPA Reference Method 19A (48 FR 48960, Oct. 21, 1983) requirements and because it may show where alternative procedures need to be incorporated or approved for use somewhere within the proposed regulation. A previous study(26) estimated that only 16% of the 90 utilities surveyed had existing sampling and analytical procedures that complied with the requirements of the proposed regulation. This section does not attempt to compare existing practices with those requirements but only identifies what the more commonly used practices are.

## 6.1 INFORMATION SOURCES

One of two major information sources was a report entitled "Electric Utility Coal Sampling and Analysis Practices: A Comparison to Proposed EPA Reference Method 19A Requirements Based on Utility Responses to FERC Survey"(26). This report tabulated and analyzed certain data collected from a survey of 190 utility plants for coal sampling and analysis information. The tabulations were set up so they could be compared with the proposed Reference Method requirements; therefore, the sampling method was not specifically defined beyond a group of ASTM methods. The sampling location was not specified other than "as-received" or "as-fired," and the method of sulfur analysis was not defined if any method other than the ASTM standards was used.

The other source of information was individual contacts at 24 utility plants that Versar had previously worked with. These plants were part of a nationwide sampling program and, as such, represent a variety of boiler types, coal types, and geographic locations.

Since information was solicited from individual contacts, more specific information was obtained than was presented in the Baker report(26). Also, a few inconsistencies between the FERC survey data and the individual contacts were found for some plants. These inconsistencies (e.g., type of sulfur analysis method used) were resolved in favor of the responses of the individual contacts.

Some of the 24 plants contacted by Versar were also included in the FERC survey. An effort has been made to display these two sources separately. Therefore, the duplicates were deleted from the FERC data base, thus decreasing the total number of plants represented by that source.

## 6.2 SAMPLING TECHNIQUES

There are several coal sampling techniques in use at different utilities. The choice of technique generally is based on either the purpose of the coal sampling program (e.g., evaluating vendor performance; monitoring coal sulfur input to boiler) or the design of the plant and adaptability of sampling equipment.

The techniques in current use are described in ASTM standard method D2234-76 (Collection of A Gross Sample of Coal) and are divided into Type I and Type II methods(6).

### 6.2.1 Type I

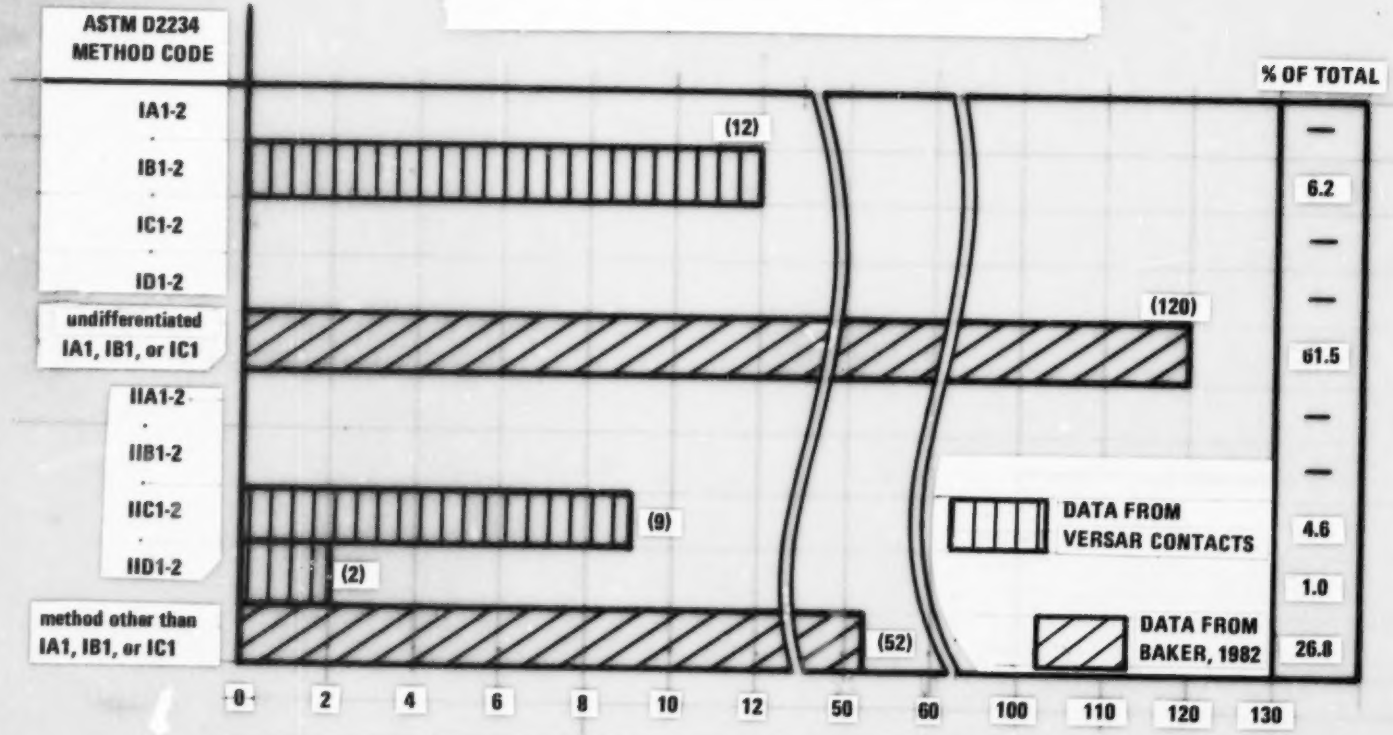
Type I methods are those not subject to discretionary bias in selecting sample increments. These methods implicitly involve some sort of automated equipment and have the code designation I. Sample increments can be removed from a stopped belt (Code A), the entire width of a moving belt (Code B), a partial width of a moving belt (Code C), or a stationary source (Code D). The sample increments can be evenly spaced (Code 1) or random (Code 2). Thus, a sample of coal composed of evenly-spaced increments taken from the entire width of a moving belt by an automatic sampling device would be designated IB1. Type I, Method B is typically used where coal is transported around a plant by belts and is the method of choice when as-fired coal quality is to be monitored. The sampling equipment can be installed at the end of a belt (e.g, where the belt changes direction). Method IA is less frequently used because it involves stopping the belt. Interruptions of this sort are not usually tolerated at electric utilities. Method IC is also less common because most automatic belt-sampling equipment incorporates a full-stream cut application in its design. Method ID1 or 2 is not commonly used.

### 6.2.2 Type II

Type II methods involve some measure of human discretion in selecting sample increments and can be used on any of the sources (Codes A-D) listed above. Increments can be evenly or randomly spaced (Codes 1 and 2) as before. Thus, manually-sampled increments taken at random times from a coal pile would be designated IID2. Type II methods are used in a wider variety of situations. For example, taking samples from the body of a truck or rail care delivering the coal and manually scooping samples from a belt inside the plant are Type II. Type II, Method D is more frequently used when coal vendor performance is being checked; Methods B and C are more common when evaluating as-fired coal quality by manual methods.

### 6.2.3 Survey Results

The sampling methods employed at several utility plants are shown in Figure 6-1. These data represent a combination of results from the Baker(26) report in which sampling methods were characterized in one of two ways (Methods IA1-IB1-IC1 or "other") they include responses from 24



NUMBER OF PLANTS (TOTAL = 195)

— DOES NOT INCLUDE ONE PLANT SAMPLING COAL WITH CYCLONES IN PULVERIZER OUTLET PIPES (MODIFICATION OF ASTMD197).

Figure 6-1. FREQUENCY OF SAMPLING METHOD

utilities that Versar had previously been in contact with. The raw data do not differentiate between even or random increment spacing. However, based on the nature of the sampling equipment and procedure in each case, it is likely that all samples taken with automatic equipment (Type I) are evenly spaced and that those taken manually (Type II) are randomly spaced, unless the utility is extremely rigorous in its application of standard methods.

Versar's experience with power plant operations indicates that stopped-belt, partial-stream cut, and stationary sampling methods are almost never used when automatic sampling equipment is in place. This is reflected in the data collected from Versar contacts, and it indicates that a large percentage of the "undifferentiated" Type I responses shown in Figure 6-1 probably refers to Method IB1. Similarly, the Versar data indicate that if Type II Methods are used, sources C and D are the most common with the emphasis on belt sampling. It can be assumed that a fairly large percentage of "other" sampling methods consist of these sources as well.

## 6.3 SAMPLING LOCATION

Coal can be sampled at many locations in a power plant depending on the method of coal handling, accessibility, and sampling objective. Most frequently, coal is sampled from a conveyor belt because this affords good accessibility and brings the coal to the sampling equipment rather than vice versa. Some operators, however, take samples from delivery vehicles to monitor incoming coal quality or from bunker storage in the plant. The most important difference between locations is whether the sample represents "as-received" or "as-fired" coal.

As-fired samples are taken at any location after the storage bunker outlet. These samples are frequently taken from the coal feeders or from a clean-out pipe on the bunker line going to the feeders. Samples are usually not taken from the pressurized lines after being pulverized, although one plant contacted did this with the aid of in-line cyclone samplers.

As-received samples are collected from any point before the bunker outlet. The main difference between as-received and as-fired samples is that the as-received samples represent coal that will be fired at some time after being retained in the bunkers or storage piles and after passing through the coal-handling system. The retention time caused by this storage as well as any blending which occurs depends on coal handling system characteristics and boiler operating parameters; it can vary significantly.

The relative frequency of sampling locations from 195 utility plants is shown in Figure 6-2.

## 6.4 BIAS TESTING/SAMPLING QUALITY CONTROL

Bias testing of sampling methods involves determining the difference (usually on a statistical basis) between samples taken by the method or equipment to be tested and those taken by some reference method. The most widely-used reference method for these comparisons is the method for collection of a gross sample of coal (ASTM D2234-76, Appendix 1)(6). Other tests have been formulated by manufacturers of sampling equipment, but these are not prevalent (see Section 3.0).

Discussions with several utility plant representatives about sampling methods used at each plant indicated that bias testing is not routinely performed, and may be done only if the plant is undergoing some sort of performance or compliance test, if at all. The utility representatives also indicated that routine quality control was not performed at most plants other than to ensure that automated sampling equipment was properly maintained and adjusted or that standard manual sampling procedures were followed during daily operations.

## 6.5 ANALYTICAL PROCEDURES

### 6.5.1 Sulfur Analysis

Several analytical methods are available for the analysis of coal characteristics, particularly for sulfur. The sulfur analytical methods

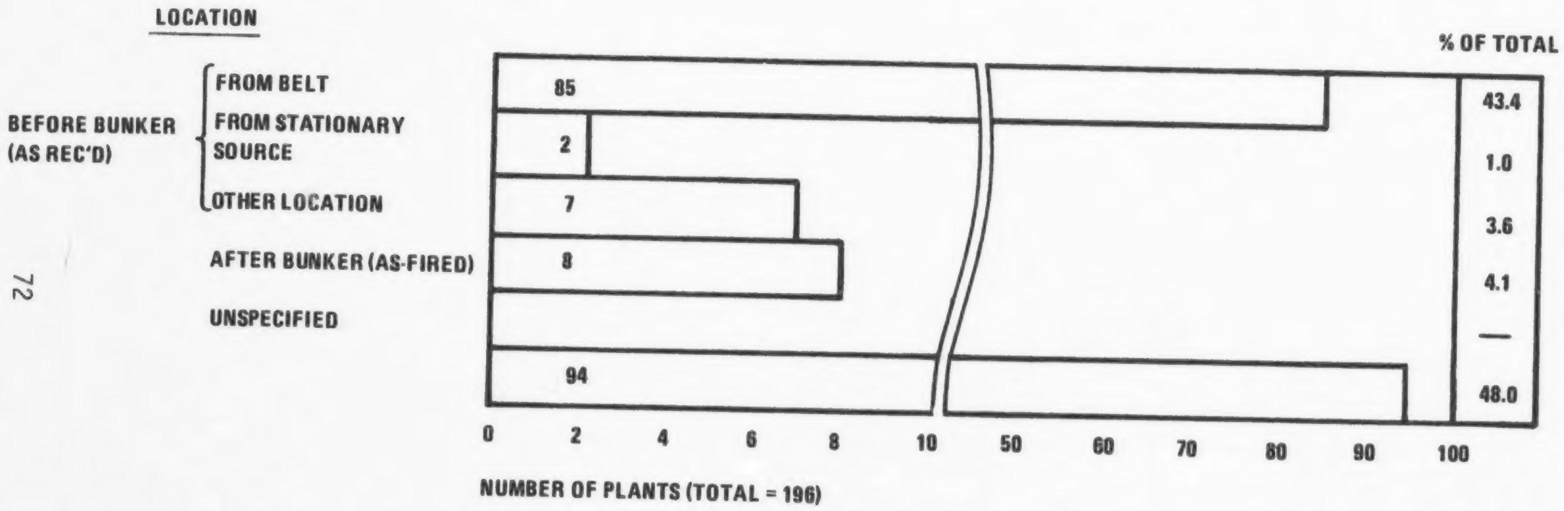


Figure 6-2. **SUMMARY OF SAMPLING LOCATIONS**

can be roughly divided into standard wet chemical techniques and automatic photometric techniques. The wet chemical techniques are generally the standard reference methods, and they are described in ASTM D3177-75, Test Method for Total Sulfur in the Analysis Sample of Coal and Coke(6). The photometric techniques are basically modifications of these standard techniques and employ colorimetric, turbidimetric, or infrared absorption to quantify the amount of sulfur evolved by the various analytical preparation procedures (usually combustion).

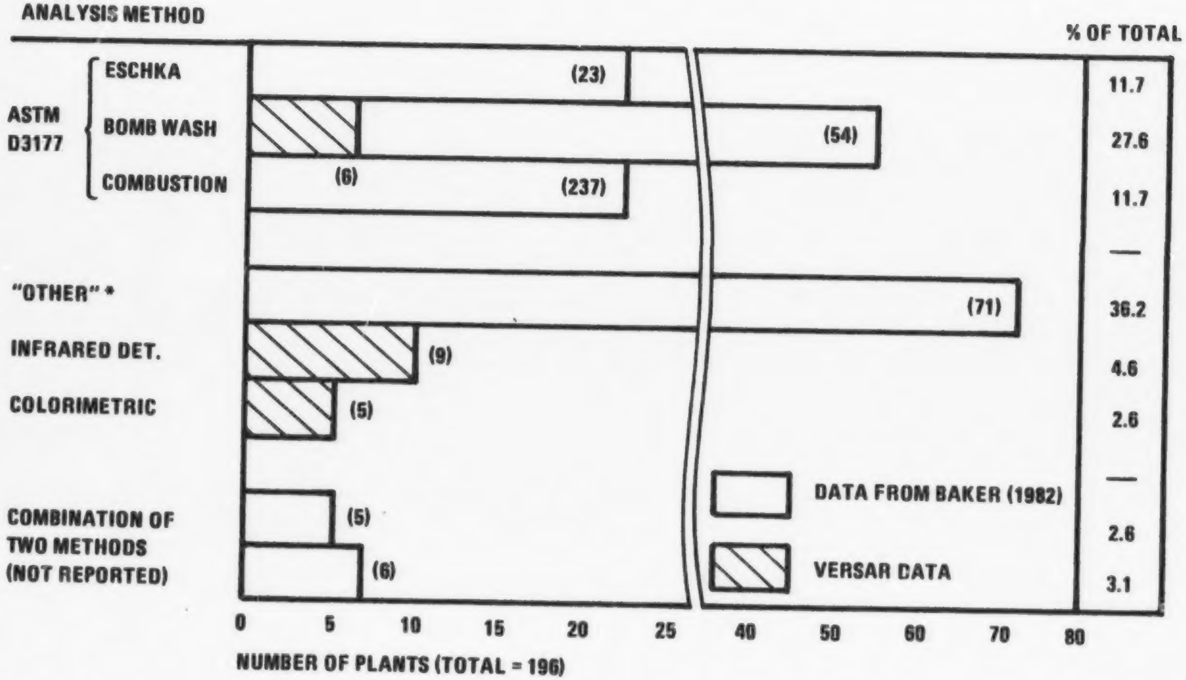
The results of the survey of sulfur determination techniques are presented in Figure 6-3. The data indicate an almost even split between standard wet chemical methods and automated analysis techniques. Note that the data from the Baker (1982)(26) report listed as "other" were assumed to refer to automated photometric techniques since the other categories listed were very specific.

### 6.5.2 Heat Content, Moisture, Ash Analysis

The heat content of coal samples is determined with either an adiabatic calorimeter ASTM Method D2015-77, or an isothermal calorimeter ASTM Method D3286(6). Nearly all the utility plants surveyed (184 out of 196, or 93.9%) determined the heat content of their coal samples with the Parr adiabatic colorimeter, using Method D2015. Four plants (2.0%) reported using the other method.

Residual moisture determinations were listed two ways in the Baker (1982) report. Those conducted according to ASTM Method D3173 or those conducted by grinding the coal to 60-mesh size but performing some test other than that specified in D3173. The results show that 85.3% of the plants surveyed reported using D3173. Another method was reported to be used by 4.1%; 20 utility plants did not list any method.

Total moisture information was derived by using ASTM D3302 on 60-mesh coal (67.4% of respondents), by a test other than D3302 on 60-mesh coal (6.8% of respondents) or by an entirely different method (23.3% of respondents); five utility plants did not report.



*"OTHER" CATEGORY IN BAKER (1982) MOST PROBABLY REFERS TO A COLORIMETRIC OR PHOTOMETRIC AUTOMATED ANALYSIS METHOD

Figure 6-3. FREQUENCY OF SULFUR ANALYSIS METHOD

Information about ash analysis was not collected from any of the information sources; however, it seems likely that since the majority of stations used ASTM standard methods for moisture and heat content analyses, a similar majority would use the appropriate standard method for the analysis of ash.

## 6.6 ANALYTICAL QUALITY CONTROL

Most of the utilities contacted indicated that they practice fairly extensive quality control procedures, ranging from analyzing standard samples of coal with each batch of regular analyses to participating in "round-robin" programs with other laboratories. The most frequent response was that standard samples are run on a daily, every-other-day, or weekly basis, often in combination with batch standards, blanks, or duplicates. Only a few utilities reported conducting QC procedures with a frequency of more than one month.

## 6.7 SUMMARY

The above discussion indicates that the following sampling and analytical procedures are common in current utility practice:

1. Sampling from conveyor belts using automated full-stream cut equipment is used by approximately 50% of all the plants considered by Versar, and probably 67% of all the plants considered in this survey.

2. Sampling from a conveyor by taking random manual grab samples is used by 38% of the plants contacted by Versar, and probably 36% of all the plants considered.

3. Sampling coal to define as-received quality is used by 43% of all plants considered as opposed to 4% taking as-fired samples; however, no location was specified for 48% of the plants.

4. Determining heat content with an adiabatic calorimeter is used by 74% of all plants considered.

5. Analyzing for sulfur by the bomb-washing method is by used 54% of those plants reporting that wet chemical procedures are used.

6. Analyzing for sulfur using automated, infrared-detector-equipped analyzers may be more prevalent than use of standard wet-chemical procedures.

7. Using ASTM standard methods for the analysis of residual moisture, total moisture, and ash is universal.

8. Calibrating equipment with standard samples, duplicates, or blanks, is done most frequently on a daily, every-other-day, or weekly basis.

9. Analyzing standard samples is done for each batch (usually 5 to 10 samples).

## SECTION 7

## THE IMPORTANCE OF COAL SULFUR PARAMETERS

### 7.1 TIME SERIES

The problem of predicting what will happen in the future is extremely important because many of the decisions we make in our everyday lives are based on predictions of conditions that are likely to exist in the future. Predictions of future events are called forecasts and the act of making such predictions is called forecasting.

Clearly, forecasting is very important in many types of organizations since the predictions of the future must be incorporated into the decision making process. For example the government of a country must be able to forecast such things as air quality, water quality, unemployment rate, inflation rate, and welfare payments in order to formulate its policies.

Since accurate forecasts are required in so many decision making forecasts, a forecaster must rely on information concerning events that have occurred in the past; that is, in order to prepare a forecast, the forecaster must analyze past data in order to identify a pattern that can be used to describe it. Then this pattern is extrapolated, or extended, into the future in order to prepare a forecast.

Time series data are the most widely used data for forecasting. A time series is a sequence of measurements of the same variable made at different times. Equally spaced time points are used in most time series studies, although they are not always used. Business time series often involve yearly, quarterly, or monthly observations, but any other time period may be used.

There are many examples of time series data, for example: sales of a particular product over time, daily mean temperature over time, and air or water quality over time. In particular, daily $SO_2$ emissions data are a time series.

## 7.2 COAL TIME SERIES

The measured values of daily sulfur content, ash content, and heating value for coal are examples of time series. We will denote by C the "true" value for the coal property being measured. Unfortunately, we do not exactly report C since the measuring process is not exact. If we let X be the reported data, then symbolically we may write

$$X = C + ME$$

where ME is the possible error introduced by the measuring process. This latter term consists of variations in the sampling and analysis procedures. It incorporates possible errors in representing the entire coal or gas stream by a single laboratory sample and in the procedures used to obtain that single sample. It also incorporates possible human errors in the analytical procedures or in reading output from automated analytic equipment. Last, it also incorporates possible equipment malfunction. We assume that the level of ME is unrelated to that of the actual process C and that, on average, ME is zero. Of course, for any particular measurement, ME probably will not be zero.

## 7.3 VARIABILITY OF COAL PARAMETERS

The variance is the average of the squares of the deviations of the observations from their mean. Therefore, the variance makes sense as a measure of spread or variability around the mean. To understand this, note that if the observations are spread out, they will tend to be far from their mean, both above and below. Some deviations will be large positive numbers, and some will be large negative numbers. But the squared deviations will all be large and positive, so the variance will be large when the data are spread out and small when the data are close together.

### 7.3.1 General Characteristics

Variability in coal is an inherent quality which is a function of the geologic and chemical processes of its formation. Coal originated from the deposition of plant materials in swampy areas at various time intervals during the last 50 to 350 million years. These depositions have resulted in more or less horizontal strata which we denote as seams. Subsequent geological activities have distorted these layers to varying degrees. Thicknesses vary from several inches to several hundred feet.

Although coals within a given seam are more consistent in their properties than coals from different seams, substantial variations in coal properties exist within larger seams both vertically and horizontally. The statistical treatment of coal data must recognize both the spatial relations (correlation) as well as the random elements of the samples.

### 7.3.2 Importance of Variability

The ability of a coal to pass emission standards depends on the mean level as well as the variance and correlation (see 7.4 below) of its sulfur content. To see the effect of the variance, let us consider the hypothetical case of a source burning 1.0 pounds of $SO_2$ per MMBtu coal with a 1.2 pounds of $SO_2$ per MMBtu standard. If the variance were zero, then each data point would be 1.0 and the source would clearly be in compliance always. However, there is variation in true data. If the data were normally distributed, then about 16 percent of the data lies above one, 2 1/2 percent above two and 0.13 percent above three standard deviations (the standard deviation is the square root of the variance) of the mean. So suppose we have a fairly typical coal: it would have, say, a standard deviation of 0.1. Then data above two standard deviations of the mean $1 + 2(.1) = 1.2$ would not be complying with the standard (see Figure 7-1). This would occur approximately 2 1/2 percent of the time. If the coal were more uniform (say from a dedicated mine), it might have a standard deviation of, say, 0.07. This uniform coal would be in

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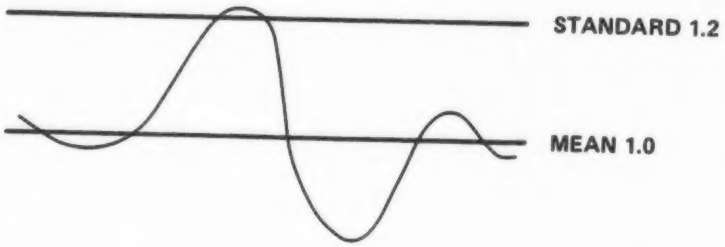

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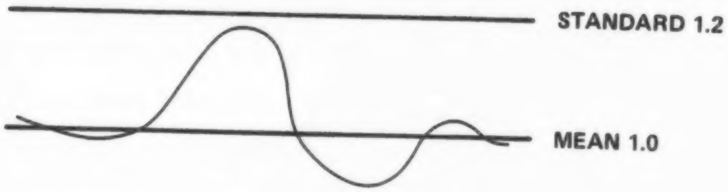

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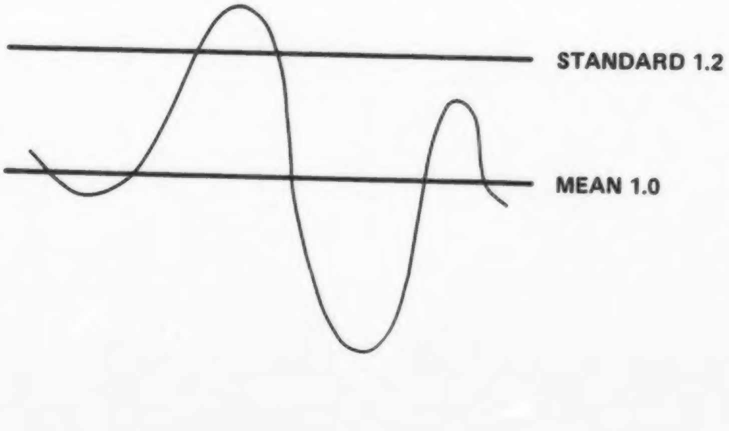


FIGURE 7-1. EFFECT OF VARIANCE ON $SO_2$ EMISSIONS

non-compliance about 0.1 percent of the time since 1.2 is about three standard deviations above the mean. Finally, if the coal were purchased on the spot market, it might easily have a standard deviation of 0.2. In that case, it would be in non-compliance about 16 percent of the time (1.2 is only one standard deviation above the mean).

### 7.3.3 Reducing Coal Sulfur Variability

Reduced sulfur variability is desired for an enhanced probability of complying with emissions standards. Processes such as coal cleaning, blending, and mixing all contribute to reducing variability.

Physical coal cleaning is a process where the coal is crushed so mineral impurities can be released. The impurities are washed out by specific gravity separation. Although originally intended only to produce a lower ash coal, the process has proved to be a good sulfur reduction step (removing a reasonable percentage of the pyritic sulfur) and a good method of producing a more homogeneous product.

The data from the EPA-Versar sulfur variability study(27) can be used to show the reduction in both mean and variance of coal properties by coal cleaning. The study used the same sampling procedures, sample preparation procedures, and laboratory analysis methods for both raw and cleaned coal samples from R & F Coal Company and Republic Steel preparation plants. Figures 7-2 and 7-3 illustrate the data.

Studies have shown that any type of coal handling operation introduces mixing of the coal which in turn decreases the variability. Naturally occurring mixing takes place in crushers, silos, and holding bins. Block shuffling takes place whenever successive truckloads, railroad loads, or silos are processed in a different order than the order in which they were received.

### 7.3.4 Measuring Error Variability

The total variance of the coal data may be split into two pieces: one due to the inherent variability of the coal and the other due to the

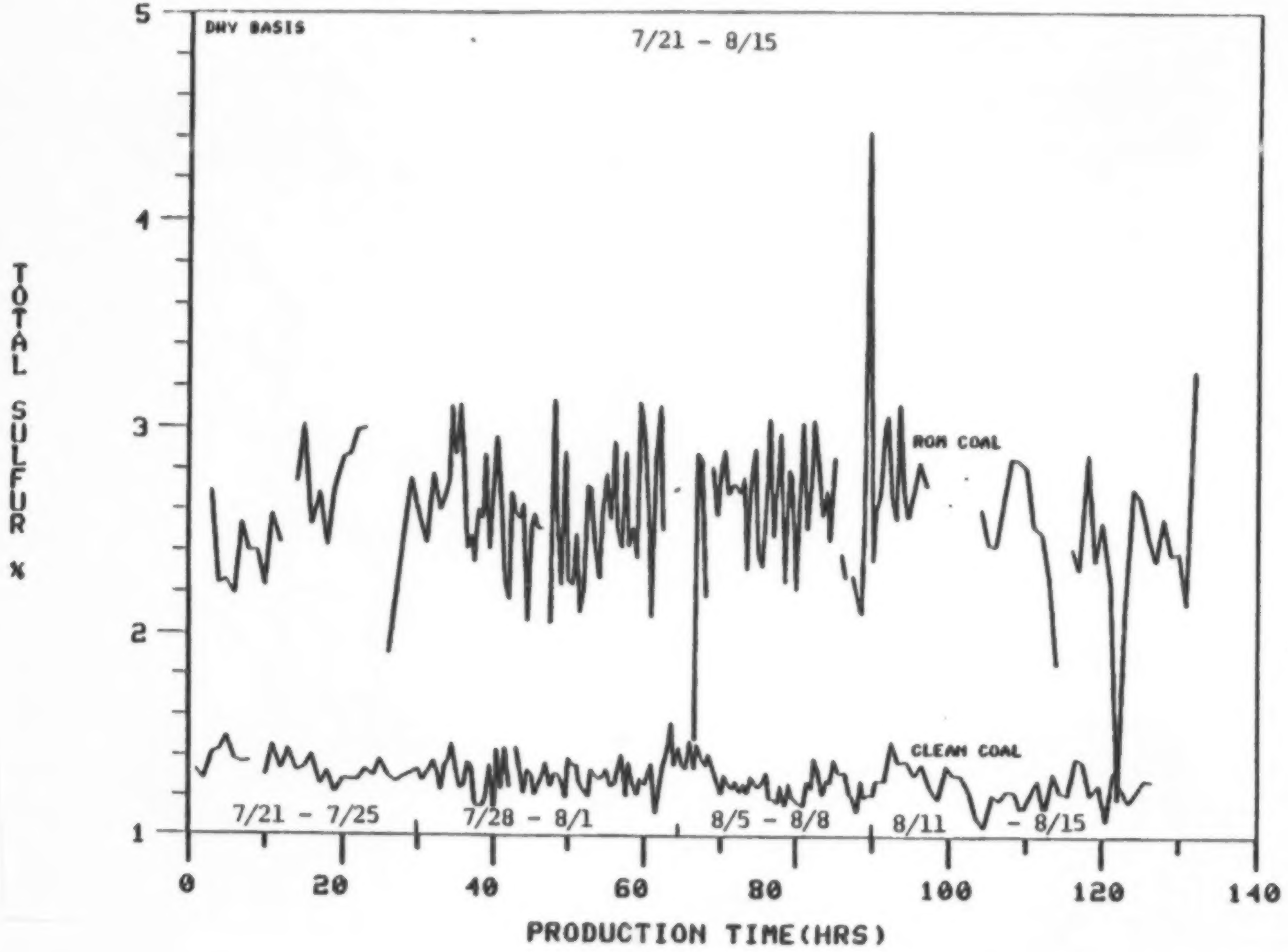


FIGURE 7-2. REPUBLIC STEEL CORPORATION - hourly increment data for total sulfur.

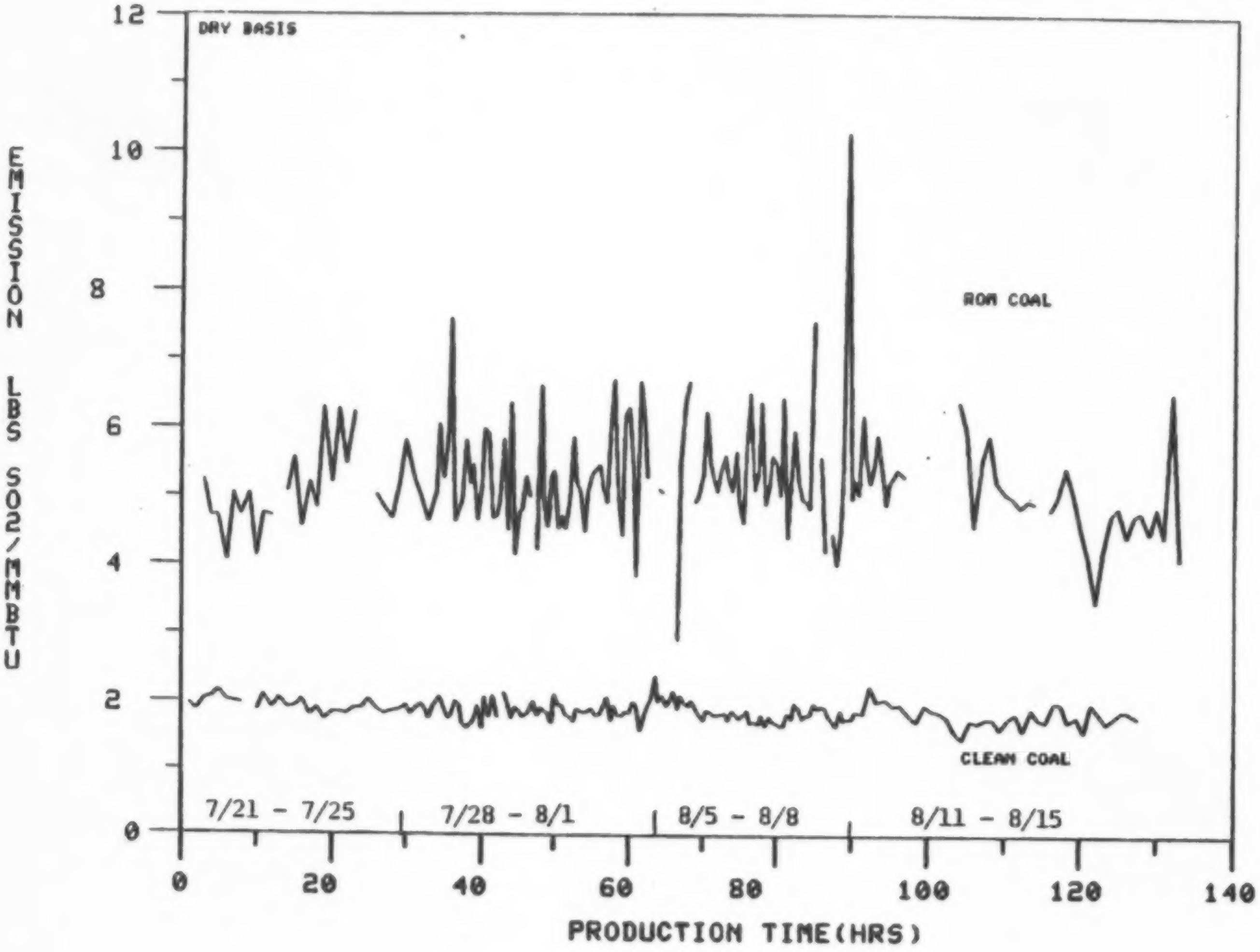


FIGURE 7-3. REPUBLIC STEEL CORPORATION - hourly increment data for sulfur dioxide emission rate.

measuring process. The measuring error variance may possibly be reduced by better QA/QC procedures, more increments per composite, additional composites, better equipment, more representative samples, etc. Alternatively, CEM and Method 6B emission measurements have lower error than does CSA. The impact of measuring variance may be seen in Figure 7-4.

## 7.4 AUTO-CORRELATION OF DAILY MEASUREMENTS

Before discussing this important parameter, a general description of correlation is provided.

### 7.4.1 Correlation

A set of data, summarized by a measure of center and a measure of spread, is far from complete. We are usually interested also in the connection between the data. Such a connection, or association, is measured by the correlation coefficient. Correlation is almost as common as mean and variance in summaries of data.

Correlation between two variables means a systematic connection between changes in one variable and changes in the other. When an increase in one variable tends to be accompanied by an increase in the other, the variables are positively correlated (associated). When an increase in one variable tends to be accompanied by a decrease in the other, the variables are negatively correlated. For example the amount of coal burned and the outside temperature are correlated. In summer in the south, as the temperature goes up more electricity for air conditioning is required. Thus, the relationship between temperature and the amount of coal used are positively correlated. Similarly, in winter in the north the relationship between the temperature and the amount of coal used would be negatively correlated since the amount of coal used increases as the temperature decreases.

It is often desirable to measure the correlation of two variables relative to their standard deviations. The correlation coefficient "r" is this measure (covariance of the two variables divided by the product of their standard deviations). The correlation coefficient is positive when an increase in the value of one variable is on the average matched

RSD = Standard Deviation ÷ mean value

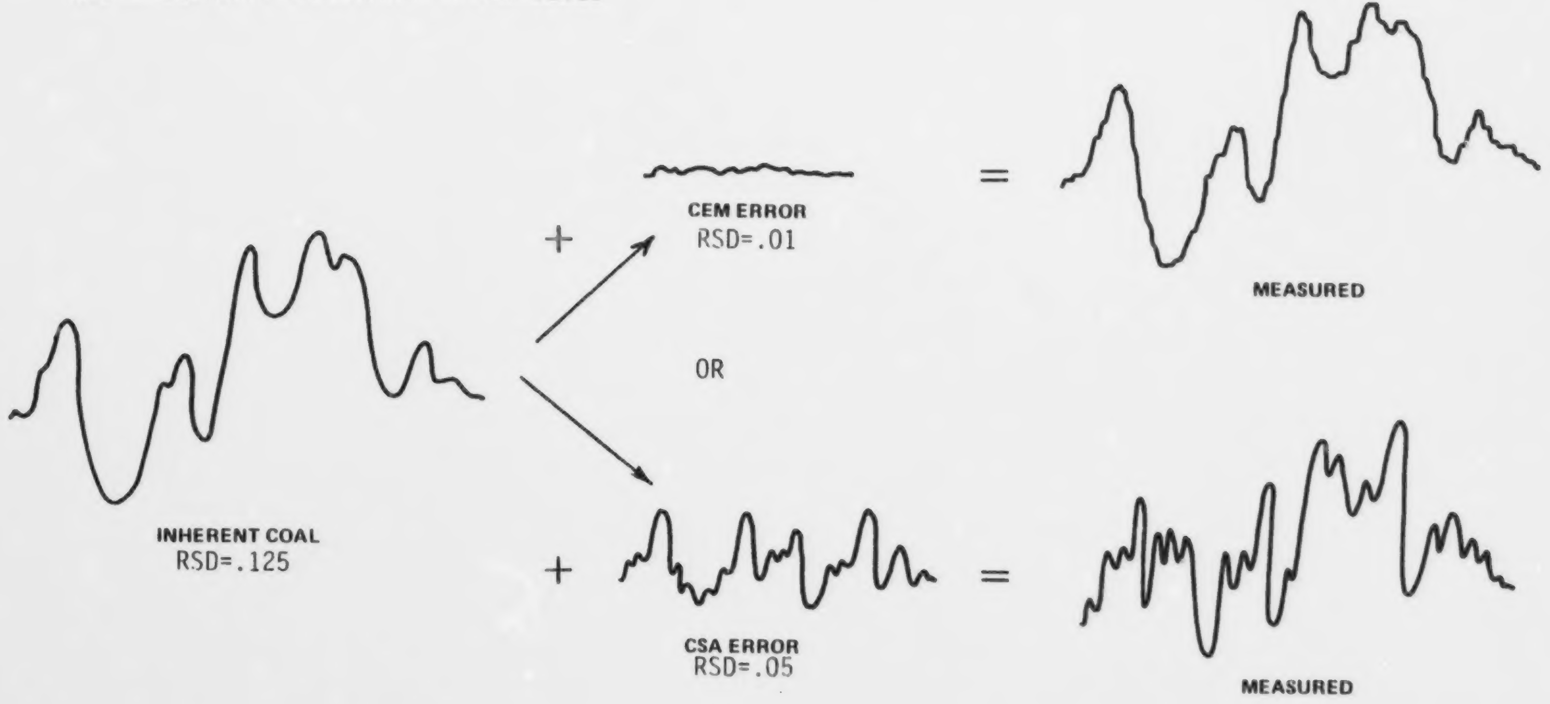

INHERENT COAL
RSD=.125
+
CEM ERROR
RSD=.01
=
MEASURED
OR
+
CSA ERROR
RSD=.05
=
MEASURED

FIGURE 7-4. VARIABILITY OF DATA

by a related increase in the other variable. It is negative when an increase in one variable is on the average matched by a decrease in the other variable.

The correlation coefficient, which is often called the "correlation," always has a value between -1 and +1. In particular, $r = -1$ means that all of the data points fall exactly on a straight line having negative slope; $r = +1$ means that all of the data points fall exactly on a straight line with positive slope. Correlations near either +1 or -1 indicate that the points fall close to a straight line. When $r > 0$ the scatter plot shows a trend from lower left to upper right and the line about which the points cluster has positive slope. For $r < 0$, the trend is from upper left to lower right and the slope is negative.

### 7.4.2 Auto-Correlation

Auto-correlation occurs when the value of a single variable is related to one or more values of the same variable. The statistical properties of a time series are measured by the autocorrelations between observations. An autocorrelation of order one is the correlation between the time series observations which are separated by one time unit. An autocorrelation of order two is the correlation between the time series observations separated by two time units. Similarly auto-correlations of higher order can be defined. All the auto-correlations are between -1 and +1.

An auto-correlation of order one close to one indicates that observations separated by one time unit have a strong tendency to move together in a linear fashion: high values tend to be followed by high values, low by low.

### 7.4.3 Auto-Correlation in Coal

The geological formation of a coal seam contributes to a spatial structure continuity in the coal mine. This structural continuity is somewhat preserved during mining, transport, and handling of coal and is transformed into a temporal continuity in the coal entering the plant. This implies that any sample value is dependent on those of the neighboring points (in time) and is correlated in some fashion. Strong positive correlation indicates that past data provides much useful information in predicting future events. In other words, a higher sulfur value at one point is more often followed by a high sulfur value at the next point.

Failure to account for the correlation structure in coal data results in gross underestimation of emission violations of actual coal(28). Consider the data given in Figures 7-5 and 7-6. The mean, the variance, the standard, and the averaging time for both data sets are about the same. Figure 7-5 shows the effect of the high correlation, 0.7 while Figure 7-6 shows the effect of a low correlation, 0.1. In both diagrams, the number of observations exceeding the standard are about the same, but with a high correlation they group together while for a small correlation they appear randomly. Hence, the average emissions over the "long-term" compliance period will not exceed the standard in Figure 7-6 while it clearly will in Figure 7-5. Thus there is an exceedance of the emissions standard for the data from Figure 7-5 while there is no exceedance indicated in Figure 7-6.

In general, the larger the correlation the harder it is to pass the emissions standard. (Blending is one effective procedure for lowering the correlation.)

## 7.5 VARIANCE AND AVERAGING PERIOD

For compliance coals leading to the same mean level of sulfur dioxide emissions, the longer the averaging period for compliance the lesser the effect variance has on the likelihood of compliance. Clearly long averaging periods allow periods where the emissions are below the standard to balance those where they are above (see Figure 7-7). Alternatively, the series of averages has a variance which decreases as the averaging period gets longer (see Figure 7-7).

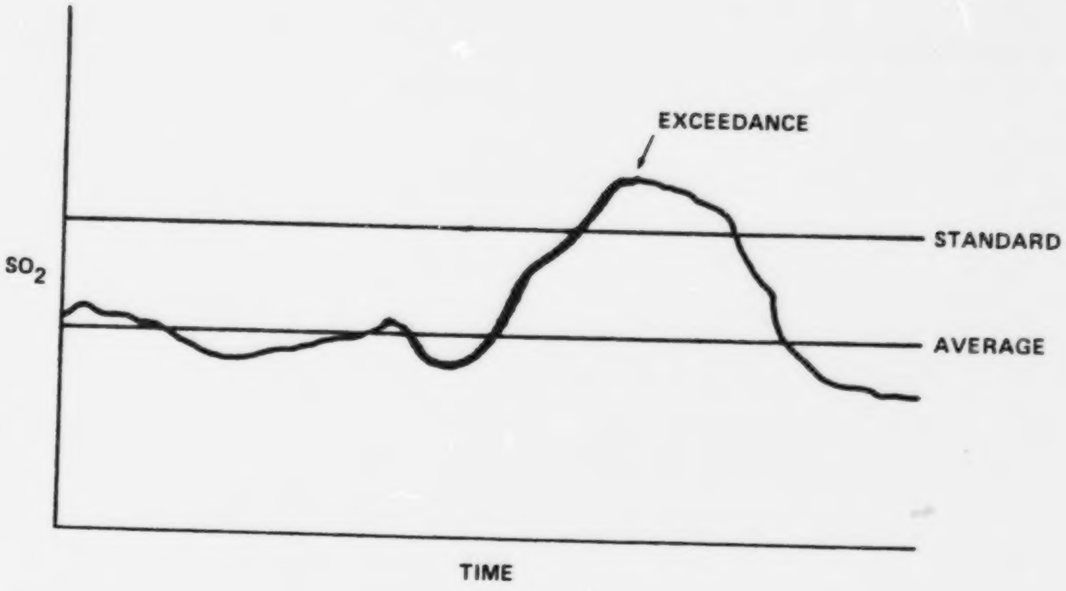


FIGURE 7-5. COAL DATA WITH r = 0.7. EXCEEDANCES EXIST

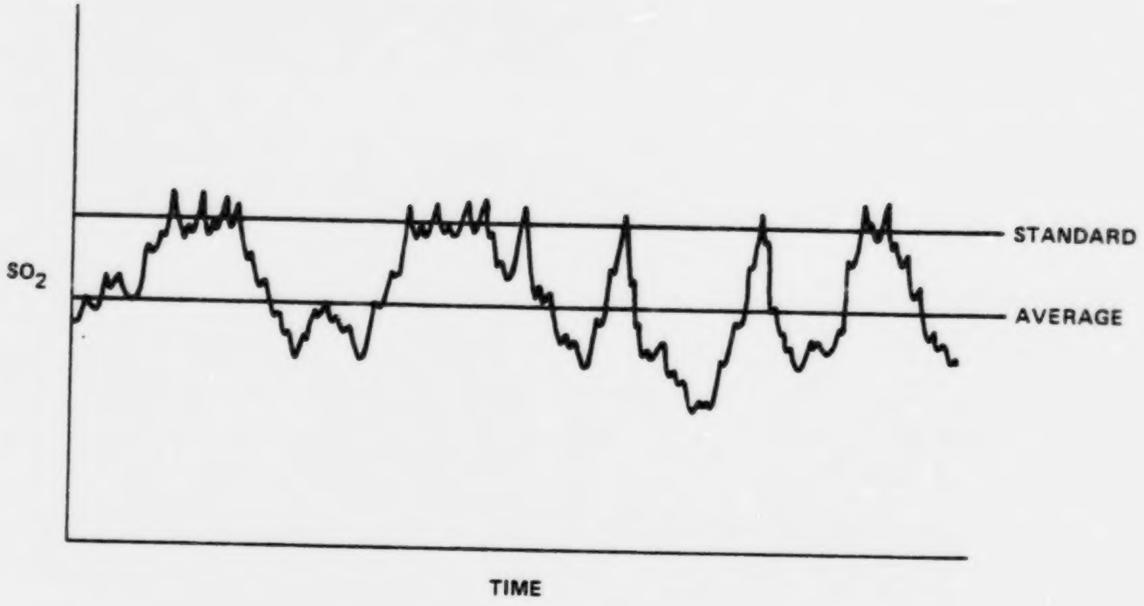
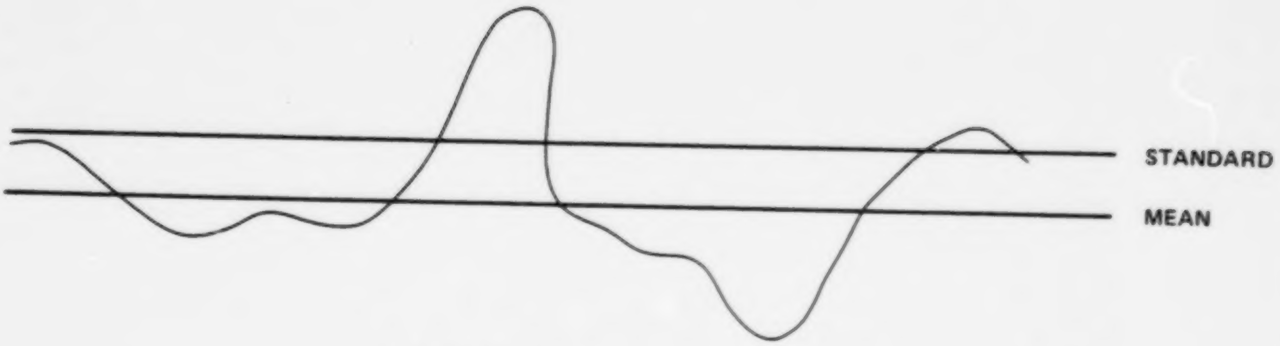
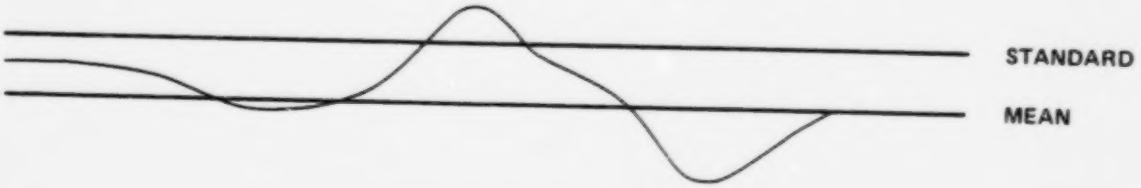


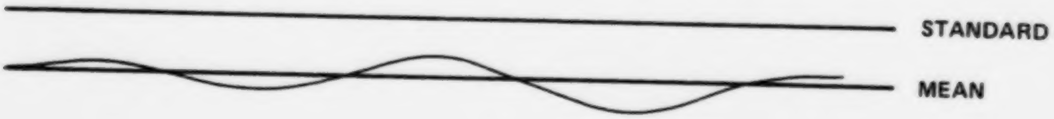
FIGURE 7-6. COAL DATA WITH $r = 0.1$. NO EXCEEDANCES.



DAILY DATA



WEEKLY AVERAGES



MONTHLY AVERAGES

*Time scales on weekly and monthly averages expanded to preserve same space relationship as daily averages.

FIGURE 7-7. EFFECT OF AVERAGING PERIOD OF COMPLIANCE AND VARIANCE

# SECTION 8

# THE MODEL

The practical application of a coal sampling and analysis (CSA) program is influenced by several factors. Among these is the adoption of a statistical theory that allows a small data set to represent a larger population. A certain amount of information pertaining to the population, or coal supply, must be known so that a small data set, or the average of a small number of increments taken in one day, can represent the entire daily coal supply. The mean, variance, covariance, and correlation are useful parameters for describing a series of coal characteristics. This information applied in an appropriate model allows an estimation, or forecast, of coal characteristics.

This section presents a statistical model which incorporates the parameters of an auto-regressive model of order one (AR(1)) and the parameters of an auto-regressive moving average (ARMA(1, 1)) model to allow an estimation of coal characteristics. The input to this model is based on measured data, for example, lbs $SO_2$/MMBTU (calculated through CSA). The model would then estimate the actual lbs $SO_2$/MMBTU in the coal supply. The input to the model can also be based on measured data of the stack emission; the output would be an estimate of the actual stack emission in lbs $SO_2$/MMBTU. In both cases the model provides an estimate for the actual measure of the coal characteristic as opposed to the measured value. The measured value (data) equals the actual value plus the measurement error. The measurement error consists of sampling/analytical errors. The actual value of the $SO_2$ emission parameter is not constant as the coal property has some inherent variability.

The focus of this section is on the application of a statistical model to a CSA program. Guidelines to minimize the measurement error are discussed, including considerations of sampling, sampling location, lot size, and analysis. In addition, it is shown how the model can be applied to stack monitoring programs, specifically continuous emission monitoring (CEM) and Method 6B (continuous bubbler (CB)).

As a prelude to this, brief descriptions of the operations of CEM and CB systems are presented.

## 8.1 EXPLANATION OF A CEM SYSTEM

Continuous emission monitoring can be done by an extractive method or an optical method. In the extractive method a gas sample is extracted from a sampling point in the stack or emission exhaust duct over a specified time period. The gas stream is sampled and analyzed continuously except for short, periodic blow backs which clear the sample line. An $SO_2$ analyzer continually measures the $SO_2$ concentration in the gas sample.

The optical method of CEM uses a light source to measure $SO_2$ levels. The light source passes through the stack and the degree of scattering of the light is read by a detector. The $SO_2$ level in the stack is calculated according to the relationship between the degree of light scattering and $SO_2$ concentration.

From this continual monitoring of the $SO_2$ level, hourly averages which consist of four equally spaced data points per one-hour period are generated.

## 8.2 EXPLANATION OF A METHOD 6B/CONTINUOUS BUBBLER MONITORING SYSTEM

Method 6B, Continuous Bubbler (CB) monitoring, is an alternative to the Method 6 determination of $SO_2$ emissions. A gas sample is extracted from a sampling point in the stack. The CB monitoring system operates continuously throughout the sampling period. The $SO_2$ contained in the sampled gas is separated and collected in the sampling train. The CB method composites the entire $SO_2$ fraction collected during the sampling period. The $SO_2$ fraction is measured by chemical analysis and the emission rate for the sampling period can be determined.

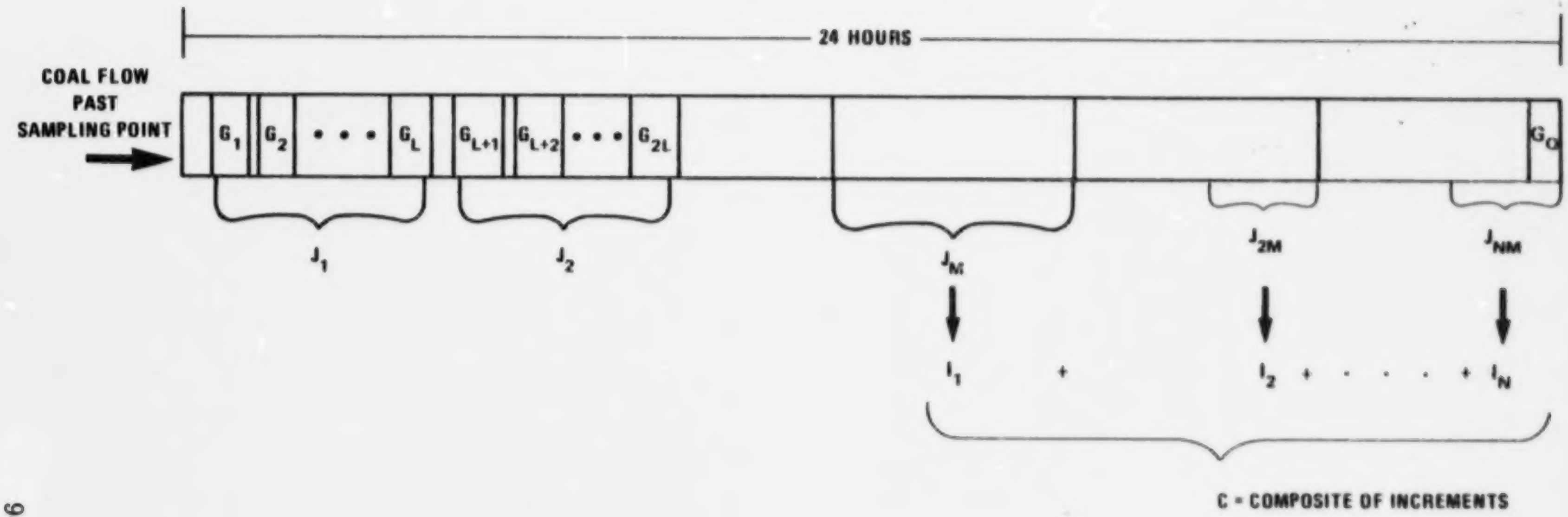
## 8.3 DESCRIPTION OF THE MODEL

The model simulates the coal properties present in either a moving stream of coal or stack emissions. In either case, the coal or stack gas passing the sampling point can be thought of as being made up of Q discrete packets of homogeneous material per day (Figure 8-1). The computer implementation is based on the assumption that Q = 1,008,000 discrete packets pass the sampling point each 24-hour period. When applied to a CSA program the numerical results are based on the assumption that the action of the sampler cutting through the coal stream takes about two seconds. Therefore, roughly 24 discrete packets would be collected with each pass of the sampler.

The actual value of the coal property in each discrete coal packet is G. For example $G_i$ can be the total sulfur contained in the ith discrete packet of coal. Each potential sampling unit is J. Thus, for coal sampling, every collection of 24 coal packets is a potential sampling unit.

The increments, I, are the actual sampling units, which correspond to the potential sampling units (J) that were actually collected. The collected increments are composited to form C as shown in Figure 8-1.

A slightly different approach is used when describing emissions data. By the time flue gases reach the stack they have become so well mixed that each unit of gas has part of the products of combustion from hundreds of the coal packets. For a CEM system, hourly emission values are based on 4 equally spaced data points. Daily averages are the average of the 24 hourly averages. In terms of our model, N=96 increments make up the daily composites (Figure 8-2). We assume that a sampling unit J is made each minute so M=15. Hence, L=Q/MN=700 coal units make up each sampling unit.


24 HOURS
COAL FLOW PAST SAMPLING POINT
$G_1$
$G_2$
$G_L$
$G_{L+1}$
$G_{L+2}$
$G_{2L}$
$G_Q$
$J_1$
$J_2$
$J_M$
$J_{2M}$
$J_{NM}$
$I_1 + I_2 + \cdot \cdot \cdot + I_N$
C = COMPOSITE OF INCREMENTS

$Q = LMN$

$Q =$ 1,008,000 discrete coal packets per 24 hour period.

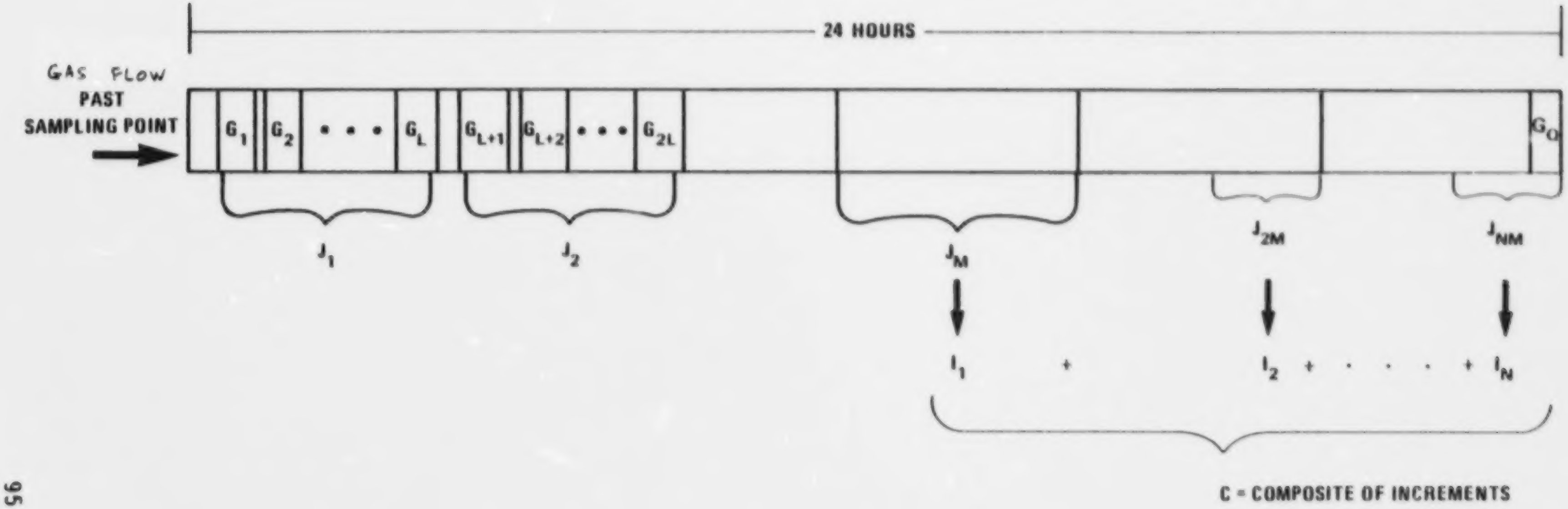
$L =$ # of coal packets collected in each potential increment (determined by coal sampler cutter speed).

$M =$ difference between increment numbers for actual sample increments.

$N =$ # of increments actually collected to form a composite.

If $L = 24$, and $N = 35$ then: $M = 1{,}008{,}000/(24 \times 35) = 1200$

FIGURE 8-1. **INCREMENT COLLECTION AND COMPOSITE IN A CSA PROGRAM**


24 HOURS
GAS FLOW
PAST
SAMPLING POINT
$G_1$
$G_2$
$G_L$
$G_{L+1}$
$G_{L+2}$
$G_{2L}$
$G_Q$
$J_1$
$J_2$
$J_M$
$J_{2M}$
$J_{NM}$
$I_1 + I_2 + \cdot \cdot \cdot + I_N$
C = COMPOSITE OF INCREMENTS

In a CEM system each potential measurement, J, is hundreds of discrete units G.
Every 15th J is selected to provide the increments, I.
The 96 I's are averaged to provide the daily composite, C.
Assuming Q = LNM = 1,008,000 we get:
L = 700 coal units measurement.
N = 96 increments per day, 4 per hour.
M = 15

FIGURE 8-2. INCREMENT COLLECTION AND COMPOSITE IN A CEM PROGRAM

The CB system is modeled in a similar manner. Since the CB system is continuously withdrawing well-mixed stack gases, it receives part of the products of combustion from each coal packet. Thus, $L=Q=1,008,000$ and $M=N=1$ (Figure 8-3).

The variability associated with the coal property G is subject to correlated and random elements. This stochastic process is modeled by an auto-regressive model of order one (i.e., an AR(1) process).

Expressed in terms of our model, the AR(1) model relates the next value of G to the current value of G using the constants $\mu$, the mean, and $\phi$, the auto-regressive parameter, via the equation:

$$G_{t+1} = \mu + \phi (G_t - \mu) + a_{t+1} \qquad (8\text{-}1)$$

where:

$G_t$ = measurement at time t,
$G_{t+1}$ = measurement at time t + 1,
$a_{t+1}$ = normal random shock at time t + 1,

and

$$-1 < \phi < 1.$$

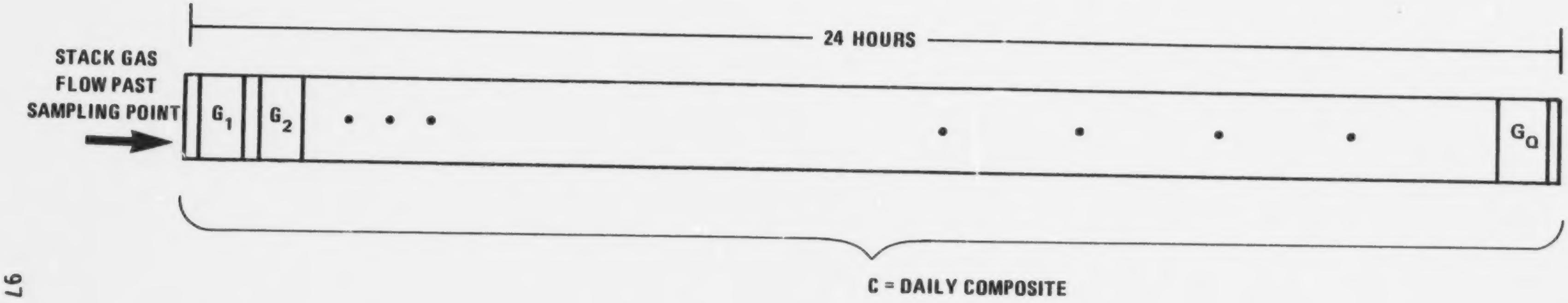
A random shock is a normally distributed random variable that is independent of all past history. It accounts for any inherent variance in the data. The auto-regressive parameter, $\phi$ , is a measure of the correlation between the current data point $G_t$ and the next value $G_{t+1}$.

If $\mu$ and $\phi$ are known, then the AR(1) model implies that the best forecast for the next data point is the mean, $\mu$, plus a multiple of the deviation of the present data point $G_t$ from the mean:

$$\text{Best forecast} = \mu + \phi (G_t - \mu).$$

It can be shown that the best forecast for the data point L units in the future satisfies:

$$\begin{aligned}\text{Best forecast for } G_{t+L} &= \mu + \phi^L \text{ (current data - mean)}\\ &= \mu + \phi^L (G_t - \mu).\end{aligned}$$


STACK GAS
FLOW PAST
SAMPLING POINT
24 HOURS
$G_1$
$G_2$
$G_Q$
C = DAILY COMPOSITE

In a CB system each measurement contains part of each discrete unit G.

FIGURE 8-3. INCREMENT COLLECTION AND COMPOSITE IN A CB PROGRAM

Hence the forecasts' dependence on the current data decays exponentially. By considering the relation (8-1), it can be shown that no future value is independent of current values. In fact, it can be shown that the correlation between any two data points L units apart is $\phi^L$. Thus, for large time lags between data points, the relationship is essentially one of independence but for small time lags it is a highly dependent one. Methods to estimate the parameters $\mu$ and $\phi$ from a data set will be discussed later. First the description of the model in the terms of a CSA program will be finished.

Assume that sampling is done periodically, J is the measured value of each potential sampling unit and its value is an average of L discrete coal packets (G) which make up each increment. Thus:

$$J_1 = \frac{1}{L}(G_1 + G_2 + \ldots + G_L)$$

$$J_2 = \frac{1}{L}(G_{L+1} + G_{L+2} + \ldots + G_{2L})$$

$$\vdots$$

$$J_{MN} = \frac{1}{L}(G_{(MN-1)\ L+1} + \ldots G_{MNL})\ .$$

Since the variability associated with the discrete coal packets is modeled by an AR(1) model, J is modeled as an average of an AR(1) model. This latter time series model is termed an auto-regressive moving average model, ARMA(1,1). Its representative is

$$J_{t+1} = \mu + \alpha\ (J_t - \mu) + b_{t+1} - \theta\ b_t$$

where

$J_t$ = measurement at time t

$J_{t+1}$ = measurement at time t+1

$b_{t+1}$ = random shock at time t+1

$b_t$ = random shock at time t

and

$$-1 < \alpha,\ \theta < +1.$$

The future value of J depends not only on the current value of J, the mean $\mu$, the auto-regressive parameter $\alpha$, but it also depends on the shock to the system at time t and an appropriate weight $\theta$ given to that shock.

If $I_1$, $I_2$, ..., $I_N$ are the actual sampled units, and $J_M$, $J_{2M}$, ..., $J_{NM}$ represent the coal property measured in the corresponding increment, then:

$$\begin{aligned} I_1 &= J_M \\ I_2 &= J_{2M} \\ &\vdots \\ I_N &= J_{NM}. \end{aligned}$$

Since I is a "thinned" version of J, it too is an ARMA(1,1) model.

Next, consider that one composite sample is formed each day from the N increments:

$$\begin{aligned} C &= \frac{1}{N}(I_1 + I_2 + \dots + I_N) \\ &= \frac{1}{N}(J_M + J_{2M} + \dots + J_{NM}). \end{aligned}$$

The composites are then used to determine the quantities that are of interest, e.g., total sulfur (weight %), so that the actual measurements in the laboratory represent the composites (C) and not the potential sampling units (J), nor the increments (I), nor the packets (G).

Again, C is an averaged value derived from the I values so C is also modeled by an ARMA (1,1) model.

In all cases the daily composite value, C, is the actual value of the coal or stack emission. It is provided by the model. A CSA program which composites the increments and analyzes the daily composite would calculate the measured value (X) of the coal. The measured value X would not equal C because X includes some unavoidable measurement error. The measurement error consists of variations in the sampling and analysis procedures.

The ARMA (1,1) model that describes the actual coal supply (C) can be extended to a model for the measured value (X) of the daily composites. Using the parameters that describe X and the properties of the model, the parameters that describe the actual coal supply can be derived.

## 8.4 ESTIMATION OF THE MEASURED DAILY COMPOSITE

To apply the ARMA (1,1) model which describes C there must be some relation between its parameters and the parameters that describe X. The value of X does not equal the actual value C because errors in the measuring process will be reflected in the value X. There are errors in the sampling, sample preparation, and sample analysis that cause the value X to be less representative of the actual coal.

The relationship between C and X is:

$$X = C + ME$$

where ME is the measurement error of the sampling and analysis procedures. ME is modelled as a normally distributed random variable that is independent of all past history and of C. The measurement error, ME, and the actual value of the composites, C, can be determined from the model once the parameters that estimate the value of the measured daily composites (X) are known. Since C is an ARMA (1,1) model and ME are independent random shocks, X is also an ARMA (1,1) model. The auto-regressive parameter of the model for X is the same as the auto-regressive parameter of the model for C. The auto-regressive parameter for X can be estimated from the data. Thus, the auto-regressive parameter for C can be estimated. Based on this value and the above model, estimates can be determined for all the parameters describing the processes G, J, I, and C.

The estimators needed to describe the daily composites, X, are (K is the number of data points):

1. estimate of mean = $\hat{\mu}$

$$= \text{average of the composites}$$

$$= \frac{1}{K} \sum_{i=1}^{K} X_i = \bar{X} \tag{8-2}$$

2. estimate of variance = $\widehat{\text{var}}\ X$

$$= \frac{1}{K-1} \sum_{i=1}^{K} (X_i - \bar{X})^2 \tag{8-3}$$

3. estimate of covariance = $\widehat{\text{cov}}_1\ X$

$$= \frac{1}{K-1} \sum_{i=1}^{K-1} (X_i - \bar{X})(X_{i+1} - \bar{X}) \tag{8-4}$$

4. estimate of correlation = $\widehat{\text{corr}}_1\ X$

$$= \widehat{\text{cov}}_1 X / \widehat{\text{var}}\ X \tag{8-5}$$

5. estimate of the second order covariance =

$$\widehat{\text{cov}}_2\ X = \frac{1}{K-1} \sum_{i=1}^{K-2} (X_i - \bar{X})(X_{i+2} - \bar{X}) \tag{8-6}$$

6. estimate of the second order correlation =

$$\widehat{\text{corr}}_2 X = \frac{\widehat{\text{cov}}_2\ X}{\widehat{\text{var}}\ X} \tag{8-7}$$

7. estimate of the auto-regressive parameter, AR(X)

$$\hat{AR}\,(X) = \frac{\hat{cov}_2\, X}{\hat{cov}_1\, X} \qquad (8\text{-}8)$$

8. estimate of the moving-average parameter MA(X):

$$\hat{MA}(X) = \frac{1}{2}\left(Y + \frac{1-C^2}{Y} \pm \right. \qquad (8\text{-}9)$$

$$\left. \pm \left(Y^2 - 2(1+C^2) + \frac{(1-C^2)^2}{Y^2}\right)^{\frac{1}{2}}\right)$$

where:

$$C = \hat{corr}_1\,(x)$$

$$Y = \hat{AR}(X) - \hat{corr}_1(X)$$

and + is used if Y is negative, - is used if Y is positive.

The estimate of the auto-regressive parameter is the basic input to the model describing G. It should be noted that equation (8-8) provides only a rough estimate of the auto-regressive parameter. A better estimator may be obtained via any Box-Jenkins Time Series routine.

The parameters of the ARMA (1,1) model which describe the daily composites (X) can be called $\alpha$ and $\beta$ with $\alpha$ being the auto-regressive parameter and $\beta$ the moving average parameter. An estimate for $\alpha$ is provided by equation (8-8), the ratio of the second order covariance to the first order covariance. An estimate for $\beta$ is also calculated through the measured data via (8-9).

The parameters of the ARMA (1,1) model which describes the actual coal (C) can be called $\alpha$ and $\delta$ with $\alpha$ being the auto-regressive parameter and $\delta$ the moving average parameter. Recall that the auto-regressive parameter for the daily composites is equal to the auto-regressive parameter for the actual coal. The moving average parameter $\delta$ for the actual coal is provided by the model.

The relationship of the parameters $\alpha$, $\beta$ and $\zeta$ for the coal data X and underlying coal properties, C, are given in the following two propositions. Their proofs may be found in (29).

Proposition 1. Let A be ARMA (1,1) with parameters $\alpha$ and $\beta$. Let B be the average of successive p elements from the series A, i.e.

$$B_k = \frac{1}{p}\,(A_{kp+1} + \ldots + A_{kp+p}) \qquad k=0,1,\ldots \quad .$$

Then B is also ARMA (1,1) with

$$\text{var (B)} = \frac{\text{var(A)}}{p} + \frac{2\ \text{cov(A)}}{p^2(1-\alpha)^2}\,((p-1) - p\alpha + \alpha^p)$$

$$\text{cov (B)} = \frac{\text{cov(A)}}{p^2(1-\alpha)^2}\ (1-\alpha^p)^2$$

kth order covariance of B = $\alpha^{p(k-1)}$ cov(B), $\quad$ k = 1,2,... .

Proposition 2. Let X = C+ME where C is ARMA (1,1) with parameters $\alpha$ and $\beta$ and ME is white noise independent of C. The X is also ARMA (1,1) with parameters $\alpha$ and $\zeta$. Also

1. cov (X) = cov (C)
2. $\text{var (C)} = \dfrac{1+\beta^2 - 2\alpha\beta}{1+\zeta^2 - 2\alpha\zeta}\ \dfrac{1-\alpha\zeta}{1-\alpha\beta}\ \dfrac{\alpha-\zeta}{\alpha-\beta}\ \text{var (X)}$
3. var (ME) = var (X) - var (C)
4. var (ME) $\geq$ o if and only if $\beta \leq \zeta$.

In the above results, var (Y) means the variance of the series Y and cov (Y) means the auto-covariance of order one of the series Y.

Now that the parameters that describe the actual coal are known the actual coal characteristic (e.g., lbs $SO_2$/MMBTU) can be calculated. The difference between the $\beta$ term in the model describing the daily composites (X) and the $\delta$ term in the actual coal (C) model measures the splitting of the variance of X into its two components: the variance of the coal and the variance of the measuring process.

## 8.5 EXAMPLE

To illustrate the use of equations 8-2 through 8-9, consider the following data set. A number of total sulfur determinations have been made on K = 30 daily composite samples from train loads of coal originating from the same mine. The variability of the data set may be attributed to the natural variability of the coal and to the measurement error. We will split this value below into its two pieces.

| Day | Total Sulfur (wt.%) |
|---|---|
| 1 | 2.46 |
| 2 | 2.64 |
| 3 | 2.68 |
| 4 | 2.49 |
| 5 | 2.31 |
| 6 | 2.38 |
| 7 | 2.46 |
| 8 | 2.97 |
| 9 | 2.68 |
| 10 | 2.50 |
| 11 | 2.76 |
| 12 | 2.50 |
| 13 | 2.45 |
| 14 | 2.65 |
| 15 | 2.73 |
| 16 | 2.55 |
| 17 | 2.51 |
| 18 | 2.45 |
| 19 | 2.30 |
| 20 | 2.26 |
| 21 | 2.39 |
| 22 | 2.64 |
| 23 | 2.33 |
| 24 | 2.38 |
| 27 | 2.31 |
| 26 | 2.52 |
| 27 | 2.31 |
| 28 | 2.46 |
| 29 | 2.65 |
| 30 | 2.52 |

Equation (8-2) gives the estimated mean of the data set

$$\hat{\mu} = \frac{1}{K} \sum_{i=1}^{K} X_i$$

$$= \frac{1}{30} (75.75) = 2.525.$$

The variance is calculated using equation (8-3)

$$\hat{\text{var}}\ X = \frac{1}{K-1} \sum_{i=1}^{K} (X_i - \bar{X})^2$$

$$= \frac{1}{30-1} (.82115) = .0283$$

Equation (8-4) gives the estimated first order covariance of the composites

$$\widehat{\text{cov}}_1\, X = \frac{1}{K-1} \sum_{i=1}^{K-1} (X_i - \bar{X})\,(X_{i+1} - \bar{X})$$

$$= \frac{1}{30-1}(0.1526) = .00526.$$

The first order correlation of the composites is simply the first order covariance divided by the variance:

$$\widehat{\text{corr}}_1\, X = \widehat{\text{cov}}_1\, X / \widehat{\text{var}}\, X$$

$$= .00526/.0283$$

$$= .185868.$$

The second order covariance is estimated using equation (8-6)

$$\widehat{\text{cov}}_2\, X = \frac{1}{K-1} \sum_{i=1}^{K-2} (X_i - \bar{X})\,(X_{i+2} - \bar{X})$$

$$= \frac{1}{29}\,(.02545)$$

$$= .0008776.$$

The second order correlation of the composites is the second order covariance divided by the variance

$$\widehat{\text{corr}}_2\, X = \widehat{\text{cor}}_2 X \,/\, \widehat{\text{var}}\, X$$

$$= .03099.$$

Finally, the ratio of the covariances gives the auto-regressive parameter:

$$\widehat{\text{AR}}\,(X) = \frac{\widehat{\text{cov}}_2\, X}{\widehat{\text{cov}}_1\, X}$$

$$= .16675.$$

The next step is to split the total variance of the daily composites .0283 into two parts: the variance of the underlying coal and the variance associated with the measuring process. Since the auto-regressive parameter for both the true coal and the measured data is the same, we estimate the auto-regressive parameter for C to be 0.16675.

In order to split the variance, we must compute the moving average term for the measured data X. Since the correlation 0.1859 is larger than the AR parameter 0.16675, we use (8-9) with a + sign to obtain

$$\widehat{MA}\ (X) = -.0198.$$

Using this, we find:

$$\text{var}\ (C) = .0145 = 51.3\%\ \text{of total variance}$$

$$\text{var}\ (ME) = .0138 = 48.7\%\ \text{of total variance.}$$

Hence, about half of the observed variation in the data is due to the inherent variability of the coal while the other half is due to the imprecision in the measurement process.

# SECTION 9

# CONSIDERATIONS OF MEASUREMENT ERROR

The previous section indicated that the actual characteristics of a coal stream could be specified knowing the measured value and the measurement error. The measured value will be more representative of the actual coal if the measurement errors in the sampling and analysis procedures are minimized.

This section discusses the considerations of sampling error, sampling location, sample lot size, sampling system performance, sample preparation, and sample analysis which contribute to the total measuring process error. Also, some elements of a quality control program which may minimize the measurement error are discussed.

## 9.1 Sampling Error

The sampling error is by far the largest component of the total error. Generally 80 percent of the total error is due to some aspect of sampling(17). Sampling error consists of random error and systematic error. Random error is caused by some chance occurrence in the sampling process. The flow of coal to a sample cutter may vary so that increments of varying weight are inadvertently collected. Random error is also related to the inherent heterogeneity of coal. Successive increments may exhibit great differences in size or chemical composition. Random error generally occurs in both positive and negative directions around the mean and tends to have an average value of zero.

Systematic error, also called sampling bias, is an error in representativeness usually caused by inaccuracies in the sampling mechanism. For instance, the geometry of a sample cutter passing through a stream may consistently cause the larger particles to be omitted from the collected sample. Another example is a sample cutter whose dimensions are not large enough for the entire width of a stream. As a result a portion of the coal stream is consistently omitted. Both of these examples illustrate the non-random bias due to sampling. Because bias is a repetitive error, as opposed to random, the results tend to be consistently higher (or consistently lower) than results from an unbiased sample.

In a CSA program an increase in variance caused by random error can mean a broader range of data around the mean emission, and therefore a greater probability of exceedances. There are several ways to minimize the potential increased variance due to random error. The ASTM approved sampling methods and techniques discussed in Section 3 are designed to decrease random error. For instance, a sampling method that takes a relatively large number of increments lessens the random error. Examples from charts will follow.

When the systematic error is considered, the bias present in the sampling mechanism must be known. A widely recommended test for bias is a statistical comparison of the data from coal collected by the automatic mechanism and of the data from coal collected by the ASTM stop-belt technique(6). Statistical tests may determine whether a bias exists and the significance of the bias. Considerations of sampler bias are further examined in Section 10.

## 9.2 Sampling Location Considerations

The goal of a CSA program to predict $SO_2$ emissions should be to attain the most accurate relation between the coal sampled and the resultant $SO_2$ emissions from that coal. It is important that the actual stack emissions can be directly related to the coal data from a specific time period. Sampling coal feedstock from an as-fired location immediately prior to its entering the boiler ensures that the stack emission is a result of that lot of coal.

Another aspect of the as-fired location which must be considered is the number of feed lines to the boiler. Withdrawing a sample from one of many feed line may not accurately represent the total coal flow into the boiler. Sampling from every feed line will reduce the uncertainty of the coal data.

Sampling from locations further from the boiler results in a need for lag time calculations to predict when the lot of sampled coal enters the boiler. For example, when coal is sampled as it is delivered to the storage bunkers, the lag time is a function of the bunker capacity, feeder operation, and feed rate to the boilers. The lag time calculation assumes that the lot of coal represented by the sample moves as an unchanging mass toward the boiler. Instead several variables may affect the flow characteristics of the coal after it has been sampled and may subsequently affect how well the sampled coal represents the predicted $SO_2$ emissions.

Coal processing and handling devices such as pulverizers or storage bunkers change the flow characteristics so that samples taken upstream of these devices tend to be only an approximation of the flow characteristics of the coal entering the boiler. The coal undergoes diffusive mixing where adjacent lots of coal are handled together, and block shuffling where the sequence in which lots of coal are handled is changed. As a result, the predicted $SO_2$ emissions for a certain time period may be based on a lot of coal that does not enter the boiler during that time period. Also, the coal may be so thoroughly mixed with other lots of coal that the coal data obtained at the upstream sampling point is meaningless.

The as-received sampling point is even more affected by lag time problems. In the case of rail shipments, samples may be withdrawn from each rail car as it is emptied into the unloading hopper, or may be withdrawn from the conveyor belt below the hopper. A CSA program that relies on coal data from samples taken as-received must take into account the fact that a trainload of coal is generally divided into a supply for plant use, and another portion which is diverted to the storage pile or coal reserve containing as much as a 91-day supply of feedstock. Coal is reclaimed from the storage pile for nights, weekends, and plant operating periods when no coal shipments are received. The as-received incremental samples may be collected from several cars in a trainload and the composite used to generate the daily coal data. Considering that the daily trainloads come from the same mine, it is possible that the mean, variance, and covariance are relatively uniform. In that case an estimate can be made of the quality of coal going into the plant. This estimate will be subject to the uncertainties and assumptions that are present when sampling from a lag time location.

The daily coal data can also be used to estimate the coal properties of the storage pile when it is reclaimed for plant use. However, the storage pile may contain coal from many trainloads so that the consideration of more than one daily coal data set would prove more representative.

Because of the general uncertainties associated with sampling from locations that require a lag time calculation, there is less assurance that the predicted $SO_2$ emission can be accurately related to the specific lot of coal sampled.

### 9.3 Averaging Time and Lot Size

Another aspect of a CSA program that must be considered is the averaging time used to determine compliance with $SO_2$ emission standards. Averaging time may be monthly, daily, or as short as three hours.

A shorter averaging time results in a smaller lot size. Emissions based on smaller lot sizes have a higher variance than data from larger lot sizes(27). The higher variance could lead to more exceedances of the emission limit than would be seen with the same coal over a longer averaging time. The short-term effect of the variance of coal properties can cause higher peak sulfur values(21). These high $SO_2$ emissions may be high enough or long enough to cause the average $SO_2$ emission over a short time period to be above compliance levels.

### 9.4 CSA Quality Assurance/Quality Control

The purpose of a CSA program is to obtain samples which are representative of the lot of coal from which the samples were taken. The coal data from the analysis of the samples is used to estimate $SO_2$ emissions. To ensure high quality results, a quality assurance/quality control (QA/QC) program must be followed. A quality control program would include the routine checks that are a part of standard procedures, for example, duplicate analyses, split samples, and periodic calibrations.

A quality assurance program, intended as a means of evaluating the effect of the routine procedural checks of quality control, includes interlaboratory comparisons of results and independent surveys of the performance of standard methods.

The following considerations for ensuring that the entire CSA program is producing high quality results will be presented in two areas: (1) sampling and (2) sample preparation and analysis.

#### 9.4.1 Sampling

The major factor that must be considered in the QA/QC evaluation of the sampling portion of a CSA program is the performance of the sampling system. An automatic sampler reduces the factor of human discretion in sampling but does not eliminate it completely. Faulty maintenance or adjustment errors have a major effect on the final result. A maintenance program must ensure that the sampler is running properly. Records should be kept of the sampler's operation so that any system failures and down times can be noted.

Sampling personnel, besides ensuring that the sampling system is operating according to standard procedures, should also document each step of the sampling procedure by maintaining complete and accurate records. To completely document the sampling system's performance, information such as coal feed rates, sample cutter speed, weight of sample increment, number of increments per averaging period, width of sample cutter, and total lot size represented should be recorded.

In addition, the boiler operating parameters should be documented. The boiler firing rate, percentage of excess air, and generating load are all important parameters and should be recorded. In addition, any periods of operational problems or down time should be documented.

The operating parameters of any $SO_2$ pollution control devices may be included in the recording of plant operation. Any device which has an effect on the emission level of $SO_2$ should be included, such as flue-gas desulfurization units or particulate control units. A particulate control unit such as an electrostatic precipitator or a mechanical dust collector will reduce $SO_2$ emissions by removing $SO_2$ which may be collected on the surface of an ash particle. Any equipment breakdowns or malfunctions that result in a pollution control system being bypassed should be documented.

In addition to these quality control procedures, an independent observer not associated with the normal sampling operation may review the sampling procedure to assure that standard methods are being followed.

### 9.4.2 Sample Preparation and Analysis

An important QC procedure for sample preparation is periodic sieve tests on the product of the sample crusher. This allows an evaluation of the performance of the equipment and the procedures. Records should be kept, documenting each step of division and reduction in sample preparation.

The analysis of duplicate samples is recommended as a QC of the analytical procedures. Additionally, all analytical equipment should be periodically inspected and calibrated. During the analysis, accurate records should describe each step. Standard methods and procedures should be followed.

The method of division, reduction, and analysis must be consistent so that any large difference in analytical results can be attributed to the sample, and not to an error in preparation or analysis.

Quality assurance measures may include reproducibility tests, where split samples are analyzed by different laboratories using the same standard method, and the results compared.

# SECTION 10

# CONSIDERATIONS OF SAMPLER BIAS

A sampling system with a systematic error, or bias, will consistently misrepresent the actual coal in the same way. This can lead to inaccurate analytical results. To prevent this a sampling system can be bias tested to determine if it is introducing any systematic errors. Of course, random sampling errors will always be present.

To test for bias the sample collected by the sampling system must be compared to a reference or standard sample. A very accurate reference sample is a full cross-section cut from a stopped belt. The reference sample increments and the test sample increments are paired observations which are then tested for systematic errors.

The method of testing for systematic error applies statistical tests of significance to the paired increments representing the same material. The statistical tests of significance test the hypothesis that the mean of the differences between reference and sampled pairs is zero. The results are interpreted as either the absence or presence of bias at a level of practical significance.

## 10.1 Bias Test Procedures

The American Society of Testing and Materials has published a Proposed Test Method for Bias(30). In general, this method is the most detailed and controlled procedure for testing the performance of automatic sampling systems. The proposed method describes the procedures for testing for systematic error between paired observations. The recommended reference sample is a full cross-section cut from a stopped belt. This reference sample would be considered the best approach for an absolute bias determination. However, secondary reference samples allowed by the ASTM when stopped belt increments cannot be collected would provide only a relative bias determination.

It is important to ensure that each reference sample increment and its paired test sample are the same weight and are prepared and analyzed under the same conditions. In the case of two or three stage sampling systems the primary test increment is reduced to a crushed subsample. Manual techniques of division and reduction will have to be applied to the reference sample. It is possible that the test sample may require less preparation for analysis. This is a possible source of bias. It is also important to note that if a mechanical system has a bias then it is better to correct the sampling system than to compensate for it mathematically.

When conducting the final statistical analysis it is not clear, as stated by the ASTM, that the procedure of randomly excluding the excess paired observations "assures that the boundaries of practical importance and statistical significance coincide."

## 10.2 Size Bias of Automatic Samplers

The Kaiser Engineers of Pennsylvania, Inc. conducted bias tests on a Run-Of-Mine (ROM) raw coal sampler at an EPRI Coal Cleaning Test Facility(31). The bias test was performed following procedures similar to the ASTM Proposed Test Method for Bias(30). The bias test samples were collected by an automatic plate type sampler. The reference sample was collected by the stopped belt method.

The results of the Kaiser test showed a bias in the size distribution collected by the test sampler (Table 10-1). A sampler's size distribution bias is determined by measuring the percentage of each size fraction actually collected and comparing it to the size distribution of a reference sample.

The EPRI sampler appears to collect less of the larger sized coal particles (1.5 inch and larger), more of the medium sized particles (8 mesh -3/4 inch), and less of the smaller sized particles (less than 24 mesh) (Table 10-1). That is, a sample collected by the EPRI sampler is less representative of the larger and smaller particles than the reference sample. The EPRI sampler is biased relative to size distribution because it does not collect all of the fine particles and larger particles present in the actual coal stream.

## TABLE 10-1. SIZE DISTRIBUTION OF EPRI BIAS TEST DATA.

| | REFERENCE SAMPLE | | EPRI TEST SAMPLE | | |
|---|---|---|---|---|---|
| COAL SIZE FRACTION | % OF WEIGHT IN SIZE FRACTION (%) | CUMULATIVE DISTRIBUTION (%) | % OF WEIGHT IN SIZE FRACTION (%) | CUMULATIVE DISTRIBUTION (%) | RATIO OF REFERENCE TO TEST SAMPLER |
| 3" x 1 1/2" | 15.07 | 15.07 | 12.82 | 12.82 | 0.8507 |
| 1 1/2" x 1" | 16.34 | 31.41 | 16.76 | 29.58 | 1.0257 |
| 1" x 3/4" | 12.46 | 43.87 | 13.38 | 42.96 | 1.0738 |
| 3/4" x 1/2" | 13.64 | 57.51 | 14.84 | 57.80 | 1.0880 |
| 1/2" x 1/4" | 14.82 | 72.33 | 16.20 | 74.00 | 1.0431 |
| 1/4" x 8 mesh | 9.52 | 81.85 | 9.49 | 83.49 | 0.9968 |
| 8 mesh x 28 mesh | 7.01 | 88.86 | 6.72 | 90.21 | 0.9586 |
| 28 mesh x 48 mesh | 2.34 | 91.20 | 2.24 | 92.45 | 0.9573 |
| 48 mesh x 100 mesh | 1.59 | 92.79 | 1.42 | 93.87 | 0.8931 |
| 100 mesh x 150 mesh | 0.60 | 93.39 | 0.55 | 94.42 | 0.9167 |
| 150 mesh x 325 mesh | 1.20 | 94.59 | 1.04 | 95.46 | 0.8667 |
| 325 mesh x 0 | 3.74 | 98.33 | 3.63 | 99.09 | 0.9706 |

## 10.3 Sulfur and Ash Bias Simulations

Using actual coal characteristics the results of Section 10.2 may be translated into possible sulfur or ash biases for the EPRI sampler. For this simulation two representative coals were tested: Helvetia coal mine (supplying Homer City) and Lower Freeport coal, Harrison County, Ohio. For each, the sulfur, ash content, and relative weight was known for each coal size class. The relative weight for each coal size collected by the sampler can be found by using the bias figures in Table 10-1. These can be converted to sulfur and ash content for the collected coal. These latter can be compared to the actual coal to determine the sulfur and ash bias.

In more detail, consider Table 10-2. The coal in 3" x 1 1/2" size fraction contains 6.99% ash, 1.27% sulfur and represents 54.4 pounds of each 100 pounds of coal. Thus of every 100 pounds of coal, this size fraction contributes 3.803 pounds of ash and .691 pounds of sulfur. Performing this same calculation for each size fraction shows that each 100 pounds of coal contains 7.165 pounds of ash and 1.465 pounds of sulfur.

Next suppose the EPRI sampler was used to sample this coal and its operating characteristics for this coal were exactly as reported in Table 10-1. Then for every 100 pounds of coal collected by the sampler only 49.39 pounds (instead of 54.4) would be in the size fraction 3" x 1 1/2" since the sampler under-collects in this size fraction. This would contribute 3.452 pounds of ash and .627 pounds of sulfur. Performing this same calculation for each size fraction shows that each 100 pounds of collected coal contains 7.167 pounds of ash and 1.484 pounds of sulfur. Hence the relative bias in sulfur is

$$(1.484-1.465)/1.465 = 1.3\% \text{ high}$$

and the relative bias in ash is

$$(7.167-7.165)/7.165 = 0.03\% \text{ high.}$$

Repeating the calculations for the Lower Freeport coal (see Table 10-3) produced relative biases of - 0.6% low for sulfur and + 0.2% high for ash.

Table 10-2. HELVETIA COAL MINE DATA

| Coal Size | Ash Content % | Sulfur Content % | Actual Coal | | | Sample Bias | Collected Coal | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Weight (per 100 lbs) | Ash (per 100 lbs) | S (per 100 lbs) | | Relative percent | Weight (per 100 lbs) | Ash (per 100 lbs) | S (per 100 lbs) |
| 3" x 1 1/2" | 6.99 | 1.27 | 54.4 | 3.803 | .691 | 0.8507 | 46.28 | 49.39 | 3.452 | .627 |
| 1 1/2" x 1" | 7.14 | 1.52 | 10.9 | .778 | .166 | 1.0257 | 11.18 | 11.93 | .852 | .181 |
| 1" x 3/4" | 7.06 | 1.54 | 6.2 | .438 | .095 | 1.0738 | 6.66 | 7.11 | .502 | .110 |
| 3/4" x 1/2" | 7.16 | 1.72 | 7.7 | .551 | .132 | 1.0880 | 8.38 | 8.94 | .640 | .154 |
| 1/2" x 1/4" | 6.85 | 1.73 | 8.6 | .589 | .148 | 1.0931 | 9.40 | 10.04 | .688 | .174 |
| 1/4" x 8 mesh | 6.77 | 1.82 | 4.8 | .325 | .087 | 0.9968 | 4.78 | 5.11 | .346 | .093 |
| 8 mesh x 28 mesh | 7.80 | 1.84 | 4.3 | .335 | .079 | 0.9586 | 4.12 | 4.40 | .343 | .081 |
| 28 mesh x 48 mesh | 10.25 | 1.83 | 1.6 | .164 | .029 | 0.9573 | 1.53 | 1.63 | .167 | .030 |
| 48 mesh x 100 mesh | 10.82 | 2.13 | 0.6 | .065 | .013 | 0.8931 | 0.54 | 0.57 | .062 | .012 |
| 100 mesh x 150 mesh | 11.47 | 2.36 | 0.2 | .023 | .005 | 0.9167 | 0.18 | 0.20 | .023 | .005 |
| 150 mesh x 325 mesh | 12.86 | 2.58 | 0.4 | .051 | .010 | 0.8667 | 0.35 | 0.37 | .048 | .009 |
| 325 mesh minus | 14.24 | 2.74 | 0.3 | .043 | .008 | 0.9706 | 0.29 | 0.31 | .044 | .008 |
| | | | | 7.165 | 1.465 | | 93.69 | | 7.167 | 1.484 |

## TABLE 10-3. LOWER FREEPORT COAL DATA

| Coal Size | Ash Content % | Sulfur Content % | Actual Coal | | | Sample Bias | Collected Coal | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Weight (per 100 lbs) | Ash (per 100 lbs) | S (per 100 lbs) | | Relative percent | Weight (per 100 lbs) | Ash (per 100 lbs) | S (per 100 lbs) |
| 1 1/2" x 1" | 15.3 | 2.52 | 14.6 | 2.24 | .368 | 1.0257 | 14.975 | 14.681 | 2.25 | .370 |
| 1" x 3/4" | 13.0 | 2.67 | 9.7 | 1.26 | .259 | 1.0738 | 10.416 | 10.211 | 1.33 | .273 |
| 3/4" x 1/2" | 13.6 | 2.89 | 16.4 | 2.23 | .474 | 1.0880 | 17.843 | 17.493 | 2.38 | .106 |
| 1/2" x 3/8" | 11.0 | 3.04 | 11.8 | 1.30 | .359 | 1.0431 | 12.309 | 12.067 | 1.33 | .367 |
| 3/8" x 1/4" | 12.5 | 3.30 | 8.9 | 1.11 | .294 | 1.0431 | 9.284 | 9.102 | 1.14 | .300 |
| 1/4" x 7 mesh | 11.4 | 3.38 | 14.3 | 1.63 | .483 | 0.9968[1] | 14.254 | 13.974 | 1.59 | .472 |
| 7 mesh x 14 mesh | 10.6 | 3.22 | 8.3 | 0.88 | .267 | 0.9586[2] | 7.956 | 7.800 | 0.83 | .251 |
| 14 mesh x 28 mesh | 11.2 | 3.23 | 5.5 | 0.62 | .178 | 0.9586[2] | 5.272 | 5.169 | 0.58 | .167 |
| 28 mesh x 48 mesh | 11.4 | 3.12 | 2.3 | 0.26 | .072 | 0.9573 | 2.202 | 2.159 | 0.25 | .067 |
| 48 mesh x 100 mesh | 12.3 | 3.65 | 4.1 | 0.50 | .150 | 0.8931 | 3.662 | 3.590 | 0.44 | .131 |
| 100 mesh x 200 mesh | 15.6 | 5.54 | 1.4 | 0.22 | .078 | 0.9000[3] | 1.260 | 1.235 | 0.19 | .068 |
| 200 mesh minus | 19.9 | 5.38 | 2.7 | 0.54 | .145 | 0.9515[3] | 2.569 | 2.519 | 0.50 | .136 |
| | | | | 12.78 | 3.126 | | 102.002 | | 12.80 | 3.108 |

---

[1] bias used for 1/4" x 8 mesh coal

[2] bias used for 8 mesh x 28 mesh coal

[3] biases calculated on assumption that one-third of the coal in 150 mesh x 325 mesh was in the size fraction 150 mesh x 200 mesh.

These results show that bias tests for size distribution must be distinguished from bias tests for coal properties like sulfur and ash. In this limited example, using one estimation of a sampler size distribution bias and two sets of coal data, the size distribution bias introduced by the sampler did not bias the sample for sulfur or ash. Essentially, the EPRI sampler collected too few of the finer coal particles and of the larger coal particles. As these sizes represent extremes in both sulfur and ash content, undersampling them both had a compensation effect.

## 10.4 Theoretical Calculations

A more theoretical model for sulfur and ash bias can be constructed using the Rosin and Rammler equation:

$$R = be^{mX^{n}} \qquad (10\text{-}1)$$

where

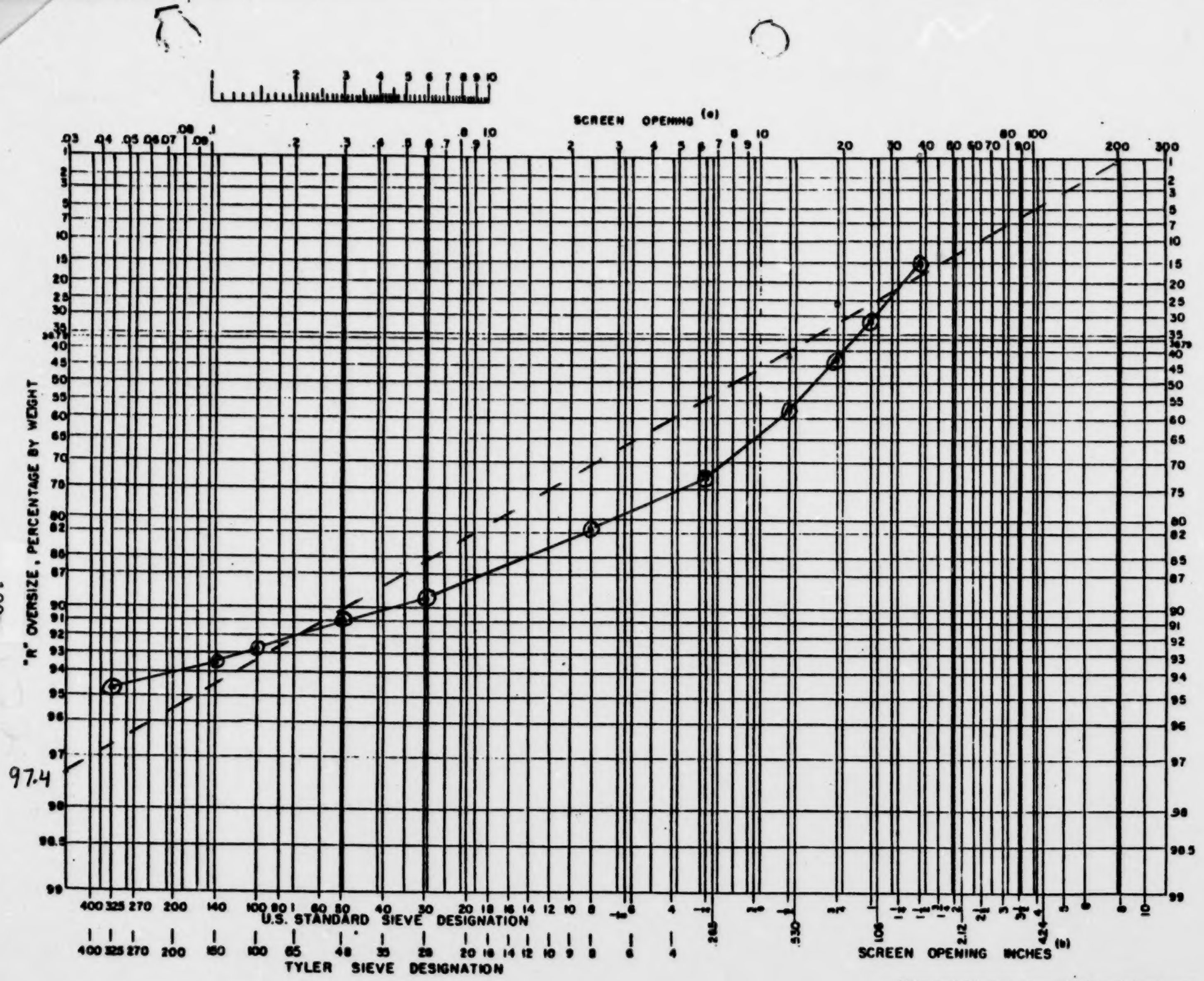
R = weight percent retained on a sieve of opening size X,
X = sieve size opening,
m, b = constants,
n = size distribution constant.

To adapt this equation to a bias test, the size distribution of the reference sample is fitted to an exponential curve (Figure 10-1). Using the EPRI data in the bias test for the run-of-mine automatic sampler (Table 10-1), the percent retained, R, and the size of sieve opening in millimeters, X, are fitted to an exponential curve to obtain:

$$R = 97.3615^{-0.046X}. \qquad (10\text{-}2)$$

Solving for X yields

$$X = \frac{(-1)}{0.046} \ln (R/97.3615). \qquad (10\text{-}3)$$



Sample No. Reference Test No. ______

Material ______

Sieve Analysis by: ______

Date ______

Plotted by: ______

Date ______

Weight of gross sample: ______

Weight of test sample: ______

Sieve Analysis:

| Point No. | Sieve Opening | Percentage Retained | Percentage Cumulative |
|---|---|---|---|
| A | 1 1/2" | 15.07 | 15.07 |
| B | 1 " | 16.34 | 31.41 |
| C | 3/4 " | 12.46 | 43.87 |
| D | 1/2 " | 13.64 | 57.51 |
| E | 1/4 " | 14.82 | 72.33 |
| F | 8 M | 9.52 | 81.85 |
| G | 28 M | 7.01 | 88.86 |
| H | 48 M | 2.34 | 91.20 |
| I | 100M | 1.59 | 92.79 |
| J | 150M | 0.60 | 93.39 |
| K | 325 M | 1.20 | 94.59 |
| | | | |
| | | | |
| | | | |
| | | | |

Characteristics:

| Point | Percentage | X, inches | Y, inches |
|---|---|---|---|
| A | | | |
| B | | | |
| | Difference | | |

Distribution coefficient, n 0.53 (0.59)

Absolute size constant $\bar{x}$ 22.5

$R = 97.36\, e^{-.046 x}$

(a) Any scale, if in millimeters, coincides with lower scale.

(b) Square hole if used as continuation of fine series.

GRAPHICAL FORM FOR REPRESENTING DISTRIBUTION OF SIZE OF BROKEN COAL

10-1

FIGURE 10-1. Size Distribution of Reference Sample.

Reference Sample

To compare the results from the reference sample data to the EPRI sampler the same equations are used. Figure 10-2 illustrates the relationship between the weight percent retained by the sampler and the sieve opening:

$$R^1 = 100.0537e^{-0.04995\ X} . \qquad (10\text{-}4)$$

Solving for X yields,

$$X = \frac{(-1)}{0.04995} \ln (R^1/100.0537) . \qquad (10\text{-}5)$$

Combining the calculated X value in each case (Equations 10-3 and 10-5) gives:

$$\left(-\frac{1}{0.046}\right) \ln \left(\frac{R}{97.3615}\right) = \left(-\frac{1}{0.04995}\right) \ln \left(\frac{R^1}{100.0537}\right) . \qquad (10\text{-}6)$$

Solving for $R^1$, the weight percent retained derived from the EPRI samplers data, yields:

$$R^1 = 100.0537 \left(\frac{R}{97.3615}\right)^{1.0859} \qquad (10\text{-}7)$$

This equation calculates for each size fraction the percentage of the reference sample that is actually being collected by the EPRI sampler.

The sulfur content, S, in each size fraction of the reference sample, R, can often be expressed by:

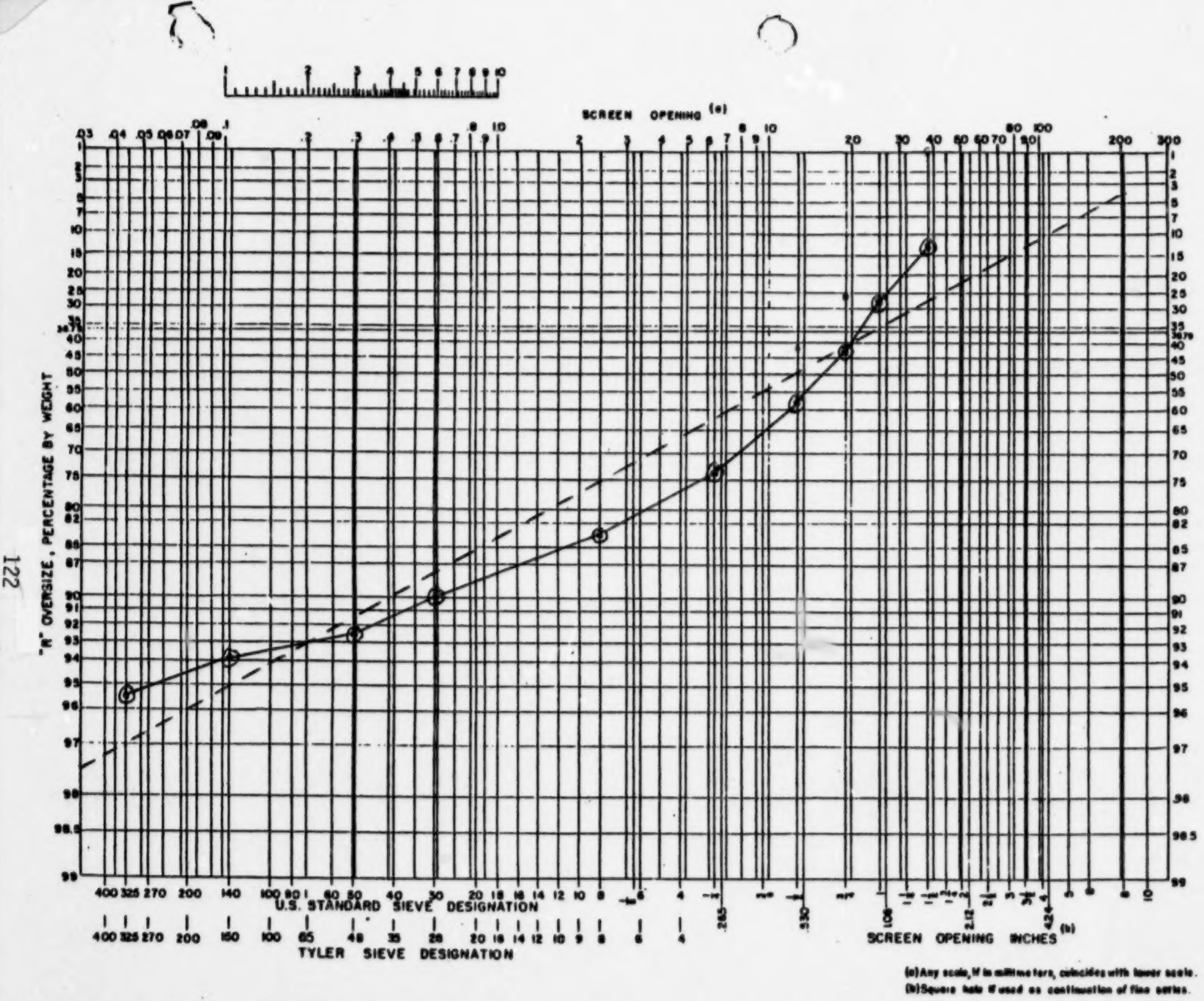
$$R = a \ln S + b \qquad (10\text{-}8)$$

where a and b are sulfur distribution constants. Similarly for the EPRI sample the sulfur content is expressed by:

$$R^1 = a \ln S^1 + b. \qquad (10\text{-}9)$$

Therefore, substituting equations 10-8 and 10-9 into equation 10-7 gives:

$$a \ln S^1 + b = 100.0537 \left(\frac{a \ln S + b}{97.3615}\right)^{1.0859} \qquad (10\text{-}10)$$



(a) Any scale, if in millimeters, coincides with lower scale.
(b) Square hole if used as continuation of fine series.

Sample No. Test Test No.
Material:

Sieve Analysis by:
Date
Plotted by:
Date
Weight of gross sample:
Weight of test sample:

Sieve Analysis:

| Point No. | Sieve Opening | Percentage Retained | Percentage Cumulative |
|---|---|---|---|
| A | 1½" | 12.82 | 12.82 |
| B | 1 " | 16.76 | 29.58 |
| C | 3/4" | 13.38 | 42.96 |
| D | 1/2" | 14.84 | 57.80 |
| E | 1/4" | 16.20 | 74.00 |
| F | 8M | 9.49 | 83.49 |
| G | 28M | 6.72 | 90.21 |
| H | 48M | 2.24 | 92.45 |
| I | 100M | 1.42 | 93.87 |
| J | 150M | 0.55 | 94.42 |
| K | 325M | 1.04 | 95.46 |
| | | | |
| | | | |
| | | | |
| | | | |

Characteristics:

| Point | Percentage | X, inches | Y, inches |
|---|---|---|---|
| A | | | |
| B | | | |
| | Difference | | |

Distribution coefficient, n 0.57 (0.56)
Absolute size constant, x̄ 22 mm

$R = 100\, e^{-.050\, x}$

GRAPHICAL FORM FOR REPRESENTING DISTRIBUTION OF SIZE OF BROKEN COAL

FIGURE 10-2. Size Distribution of Test Sample.

Solving for $S^1$ will express the sulfur content of the EPRI sampled weight fraction in terms of the reference sampled weight fraction:

$$S^1 = \exp\left(\frac{100.0537}{a}\left(\frac{a \ln S + b}{97.3615}\right)^{1.0899} - \frac{b}{a}\right). \tag{10-11}$$

To illustrate the use of equation (10-11) and the effect of sampler bias on sulfur content determinations the following data is provided (Table 10-4). This table presents the sulfur content found in various coal size fractions of a full cross-section cut of a stopped belt. This sample is the reference sample. Using this data and equation 10-11 which estimates the effect of the samplers bias on the total sulfur determination, the test sample, $S^1$, can be compared to the reference sample, S.

The first step is to fit the total sulfur content of the reference sample, S, and the corresponding size fraction, R, to a line of reasonable fit (Figure 10-3, correlation coefficient = 0.89) using a logarithmic relationship:

$$R = 93.042 \ln S + 33.253. \tag{10-12}$$

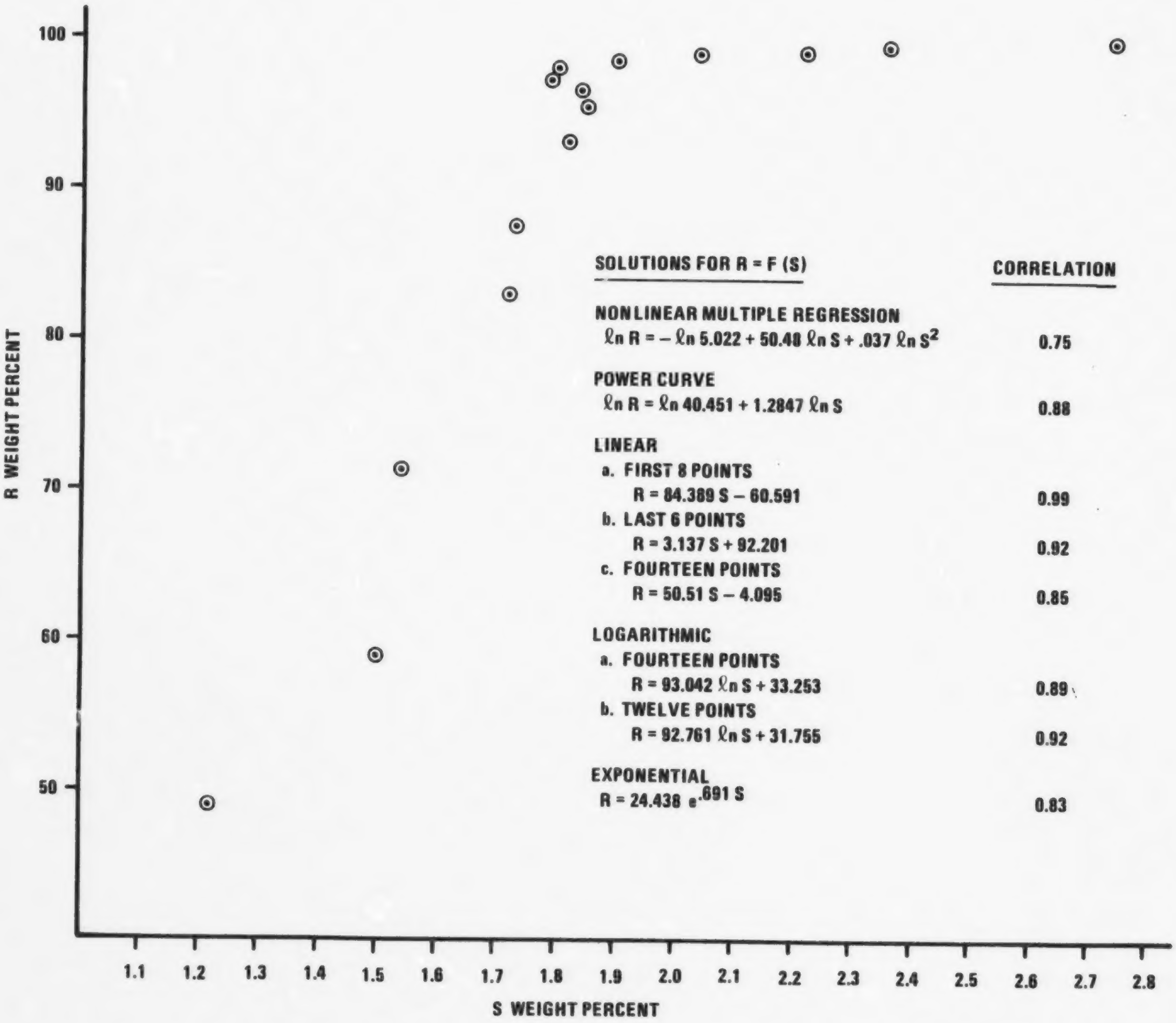
Substituting this expression describing the relationship between total sulfur and size fraction in the reference sample into equation (10-11) and simplifying gives:

$$S^1 = \exp\left(1.0754\,(0.9956 \ln S + 0.3415)^{1.0859} - 0.3574\right). \tag{10-13}$$

This equation indicates the sulfur value that would result from samples collected by the biased sampler. A range of theoretical reference sample sulfur values and the calculated sulfur values collected by the biased test sampler are presented in Table 10-5. This table illustrates how the amount of sulfur in the reference sample affects the degree of bias, and therefore the amount of sulfur, in the test sample. The bias, expressed as percent error, is more pronounced when the sulfur content is highest, at 2.8 percent. This is due to the fact that the finer particles of coal have a higher sulfur content (weight percent) than larger particles

## TABLE 10-4. SIZE FRACTION VS. SULFUR AND ASH CONTENT.

| COAL SIZE FRACTION | SIZE FRACTION (WEIGHT %) | SULFUR CONCENTRATION (WEIGHT %) | ASH CONCENTRATION (WEIGHT %) |
|---|---|---|---|
| >2" | 45.0 | 1.22 | 6.94 |
| 2" - 1 1/4" | 14.1 | 1.50 | 7.24 |
| 1 1/4" - 3/4" | 12.4 | 1.54 | 7.06 |
| 3/4" - 3/8" | 11.5 | 1.72 | 7.16 |
| 3/8" - 4 mesh | 4.8 | 1.73 | 6.61 |
| 4 mesh - 8 mesh | 4.8 | 1.82 | 6.77 |
| 8 mesh - 14 mesh | 2.8 | 1.85 | 7.71 |
| 14 mesh - 20 mesh | 1.1 | 1.84 | 8.31 |
| 20 mesh - 28 mesh | 0.8 | 1.79 | 8.86 |
| 28 mesh - 35 mesh | 0.7 | 1.80 | 10.28 |
| 35 mesh - 48 mesh | 0.5 | 1.90 | 11.24 |
| 48 mesh - 65 mesh | 0.3 | 2.04 | 10.84 |
| 65 mesh - 100 mesh | 0.3 | 2.22 | 10.79 |
| 100 mesh - 200 mesh | 0.4 | 2.36 | 11.47 |
| <200 mesh | 0.5 | 2.74 | 14.24 |



| SOLUTIONS FOR R = F (S) | CORRELATION |
|---|---|
| **NONLINEAR MULTIPLE REGRESSION** | |
| $\ell n\, R = -\ell n\, 5.022 + 50.48\, \ell n\, S + .037\, \ell n\, S^2$ | 0.75 |
| **POWER CURVE** | |
| $\ell n\, R = \ell n\, 40.451 + 1.2847\, \ell n\, S$ | 0.88 |
| **LINEAR** | |
| a. FIRST 8 POINTS | |
| $R = 84.389\, S - 60.591$ | 0.99 |
| b. LAST 6 POINTS | |
| $R = 3.137\, S + 92.201$ | 0.92 |
| c. FOURTEEN POINTS | |
| $R = 50.51\, S - 4.095$ | 0.85 |
| **LOGARITHMIC** | |
| a. FOURTEEN POINTS | |
| $R = 93.042\, \ell n\, S + 33.253$ | 0.89 |
| b. TWELVE POINTS | |
| $R = 92.761\, \ell n\, S + 31.755$ | 0.92 |
| **EXPONENTIAL** | |
| $R = 24.438\, e^{.691\, S}$ | 0.83 |

FIGURE 10-3. SIZE FRACTION VS. SULFUR CONTENT

## TABLE 10-5. BIASED SAMPLER SULFUR CONTENT

| REFERENCE SAMPLE SULFUR CONTENT (WT %) | TEST SAMPLE SULFUR CONTENT (WT %) | PERCENT ERROR |
|---|---|---|
| 1.0 | 0.977 | 2.2 |
| 1.1 | 1.078 | 2.0 |
| 1.2 | 1.181 | 1.6 |
| 1.3 | 1.286 | 1.1 |
| 1.4 | 1.392 | 0.6 |
| 1.5 | 1.500 | 0.0 |
| 1.6 | 1.609 | -0.6 |
| 1.7 | 1.720 | -1.2 |
| 1.8 | 1.832 | -1.8 |
| 1.9 | 1.945 | -2.4 |
| 2.0 | 2.059 | -3.0 |
| 2.1 | 2.175 | -3.6 |
| 2.2 | 2.292 | -4.2 |
| 2.3 | 2.409 | -4.8 |
| 2.4 | 2.528 | -5.3 |
| 2.5 | 2.647 | -5.9 |
| 2.6 | 2.768 | -6.5 |
| 2.7 | 2.891 | -7.0 |
| 2.8 | 3.013 | -7.6 |

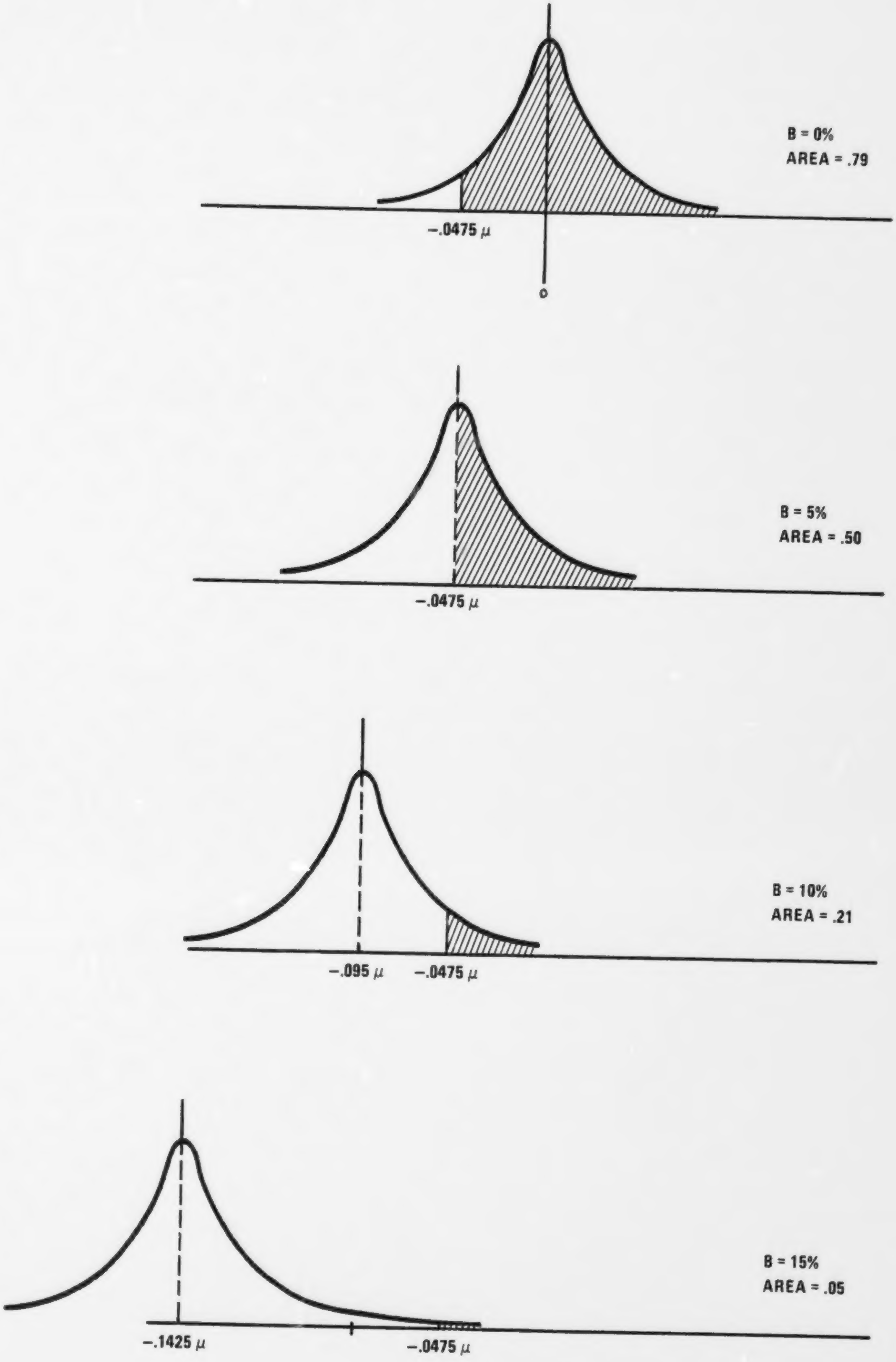


FIGURE 10-4. AFFECT OF BIAS ON RELATIVE ACCURACY TEST

(Table 10-4) and the fact that the sampling system used in this example undersamples the finer particles. The result is that the sampling system is missing the portion of the coal stream with the highest sulfur content. This bias becomes more pronounced when there are more finer coal particles in the coal stream. The opposite is true for larger coal particles.

## 10.5 Bias Testing Using Emissions Monitors

The previously described methods test for bias by automatic sampling systems by comparing the results to a full cross-section cut from a stopped belt. An alternate reference sample could be obtained from an emissions monitor (either CEM or Method 6B). Daily $SO_2$/MMBtu calculations from both methods could be compared as paired observations and the possible bias determined. However, this procedure is not as simple as the direct comparison between two coal samples. Possible complications are:

1. The unknown lag between the lot of coal which is sampled as it is bunkered and the measurement of pounds of $SO_2$/MMBtu in the flue gas corresponding to the combustion of the same lot of coal.

2. The consistency of sulfur capture mechanisms may vary with changing coal characteristics, boiler operating conditions, and pollution control device efficiency. Thus the relative difference between perfectly operating CSA and emissions monitor data due to sulfur capture is not known.

In our analysis below, we will make the following assumptions regarding these issues:

1. There is a twelve hour delay between CSA measurements and emission monitor measurements on the same coal lot. Thus the average of day t and day t+1 from the monitor represents the same coal as that sampled on day t from the CSA procedures.

2. There is a constant depletion of s% and an allowed depletion credit of d% in the CSA measurement relative to the true coal property. Note that we do not assume that s=d.

3. There is a negative bias of B% in the CSA measurements relative to the true coal property.

We use our model for the true coal lb $SO_2$/MMBtu described above, i.e. that the measurements may be split into a "true process with error" and an independent white noise representing "measurement error." From our data analysis we further assume:

4. The measurement error for CSA, the measurement error for the emmisions monitor, and the true coal process all have constant Relative Standard Deviations (RSD's), i.e.

   standard deviation/measured mean = constant.

   Let

$$\text{Diff}_t = (1-d) \times \text{Coal measurement on day } t \text{ minus Emissions measurement on day } t.$$

Then

$$\text{Expected Value } (\text{Diff}_t) = [(s-d) - B(1-d)]\mu$$

where

$$\mu = \text{true mean of the process.}$$

The relative accuracy of the CSA is measured by

$$\text{Diff}_t/\text{Emissions Data on day } t.$$

If the bias is positive, i.e. the CSA procedures are producing systematically high results, then the source is "cheating itself." If the bias is negative, i.e. the CSA procedures are producing systematically low results, then it is possible that the source may be in violation of a standard based on emission but its CSA data would not so indicate. Thus, a relative accuracy test should protect against large negative biases. We are therefore interested in the probability that the relative accuracy of the CSA methods be larger than some pre-assigned (small) negative quantity:

$$A = \text{Prob}\ (-p < \text{Diff}_t/\text{Emissions}_t).$$

Using the approximation

$$\text{Emissions}_t \text{ equals its expected value,}$$

A may be computed given values for the constants.

A relative accuracy test would compare several days of CSA data to the corresponding days for an emissions monitor. If our test period is seven days and we require that the accuracy is above -p% for at least n of these seven days, then the probability of passing such a test is

$$\text{Prob (pass)} = \sum_{j=n}^{7} \frac{7(6)..(7-j+1)}{j!} A^j (1-A)^{7-j} \quad .$$

These may also be tabulated.

To select the optimal number for n, we need to make further assumptions regarding the criteria for selection. Our assumptions are:

1. At least 85% of unbiased samplers would pass the test. The most difficult case to detect unbiased sampler is for the case s=o, i.e. there is an allowance for sulfur depletion but there is no actual depletion.

2. We select that test which minimizes the probability that a biased sampler with fixed bias B would pass the test. The value n may depend on B. The most difficult case to detect bias is for the case $s=s_{max}$, i.e. actual depletion is as large as possible.

For all of our tables below we have assumed the following values:

$d=5\%$ (depletion allowance)

$0 \le s \le 5\%$ (actual depletion)

1/12 = RSD for coal
1/20 = RSD for CSA error
1/40 = RSD for 6B error
1/100 = RSD for CEM error

.8 = correlation between daily coal measurements.

Using these values with $s=5\%$, the effect of a bias may be seen graphically. The expected value of the difference between CSA and Method 68 would be $B(.95)$ and the standard deviation can be shown to be 0.587. Using a 5% relative accuracy test relative to Method 6B, we find

$$A = \text{Prob}\,(-.05 < \text{Diff}_t/\text{Method 6B})$$

$$= \text{Prob}\,(16.1808\,(B-.05) < Z)$$

where Z is a standard normal random variable. We obtain (see Figure 10-4):

If bias is 0%, then A = .79

If bias is 5%, then A = .50

If bias is 10%, then A = .21

If bias is 20%, then A = .05.

Based on Tables 10-6 or 10-7, a reasonable test protocol would be:

1. If we wish to reject a 10% relative bias, then the CSA must pass a 10% relative accuracy test at least 5 of 7 days.

2. If we wish to reject a 15% (or larger) relative bias, then the CSA must pass a 10% relative accuracy test at least 4 of 7 days.

Relative to Method 6B, the above tests have the following characteristics (see Table 10-6). For the first test, an unbiased CSA would pass 85% of the time whereas a 10% biased CSA would pass only 23% of the time. For

## TABLE 10-6. PROBABILITIES OF CSA PASSING BIAS TEST RELATIVE TO METHOD 6B

I. Bias=0%, s=0%, d=5%

| Accuracy | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| 10% | .8002 | | | .8523 | .9668 |
| 15% | .9540 | | .9619 | .9970 | .9999 |
| 20% | .9942 | .9604 | .9993 | 1.0000 | 1.0000 |

II. s=d=5%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .2092 | | | .0057 | .0389 |
| .00 | .5000 | | .0625 | .2266 | .5000 |
| .05 | .7908 | .1933 | .5514 | .8357 | .9611 |
| .10 | .9472 | .6839 | .9509 | .9956 | .9998 |

## TABLE 10-7. PROBABILITIES OF CSA PASSING BIAS TEST RELATIVE TO CEM

I. Bias=0%, s=0%, d=5%

| Accuracy | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| 10% | .8194 | | | .8837 | .9766 |
| 15% | .9661 | | .9784 | .9988 | 1.0000 |
| 20% | .9969 | .9786 | .9998 | 1.0000 | 1.0000 |

II. s=d=5%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .1918 | | | .0039 | .0289 |
| .00 | .5000 | | .0625 | .2266 | .5000 |
| .05 | .8082 | .2251 | .5993 | .8657 | .9711 |
| .10 | .9593 | .7474 | .9696 | .9979 | .9999 |

the second test, an unbiased CSA would pass 97% of the time whereas a 15% biased CSA would pass but 4% of the time. Note that a 15% biased CSA would pass the first test only 0.6% of the time while a 10% biased CSA would pass the second test 50% of the time.

We note that our selected tests would not change if the allowed depletion were but 2-1/2% and the maximum theoretical depletion is either 2-1/2% or 5% (see Tables 10-8, 10-9). The probabilities of passing such a test would of course change. A relative accuracy test compared to Method 6B with $s_{max}$ = 2-1/2% would have the following characteristics.

Test 1. Unbiased CSA would pass 97% of the time while a 10% biased CSA would pass 23% of the time.

Test 2. Unbiased CSA would pass 99.7% of the time while a 15% biased CSA would pass 23% of the time.

If $s_{max}$ = 5%, then the tests would be less sharp and would satisfy:

Test 1. Unbiased CSA would pass 97% of the time while a 10% biased CSA would pass 52% of the time.

Test 2. Unbiased CSA would pass 99.7% of the time while a 15% biased CSA would pass 18% of the time.

## TABLE 10-8. PROBABILITIES OF CSA PASSING BIAS TEST RELATIVE TO METHOD 6B (ALTERNATIVE DEPLETION)

I. Bias=0%, s=0%, d=2-1/2%

| Accuracy | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| 5% | .6602 | | | | .8166 |
| 10% | .8923 | | .8309 | .9687 | .9964 |
| 15% | .9805 | .8714 | .9925 | .9998 | 1.0000 |
| 20% | .9981 | .9866 | .9999 | 1.0000 | 1.0000 |

II. s=d=2-1/2%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .2092 | | | .0057 | .0389 |
| .00 | .5000 | .0078 | .0625 | .2266 | .5000 |
| .05 | .7908 | .1933 | .5514 | .8357 | .9611 |
| .10 | .9472 | .6839 | .9509 | .9956 | .9998 |

III. s=5%, d=2-1/2%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .3386 | | | .0484 | .1814 |
| .00 | .6461 | .0470 | .2272 | .5233 | .7936 |
| .05 | .8782 | .4028 | .7939 | .9567 | .9943 |
| .10 | .9748 | .8366 | .9878 | .9995 | 1.0000 |

## TABLE 10-9. PROBABILITIES OF CSA PASSING BIAS TEST RELATIVE TO CEM (ALTERNATIVE DEPLETION)

I. Bias=0%, s=0%, d=2-1/2%

| Accuracy | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| 5% | .6722 | | | | .8352 |
| 10% | .9096 | | .8736 | .9804 | .9981 |
| 15% | .9871 | .9134 | .9967 | .9999 | 1.0000 |
| 20% | .9991 | .9937 | 1.0000 | 1.0000 | 1.0000 |

II. s=d=2-1/2%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .1918 | | .0003 | .0039 | .0289 |
| .00 | .5000 | .0078 | .0625 | .2266 | .5000 |
| .05 | .8082 | .2251 | .5993 | .8657 | .9711 |
| .10 | .9593 | .7474 | .9696 | .9979 | .9999 |

III. s=5%, d=2-1/2%

| Accuracy-Bias | A | n=7 | n=6 | n=5 | n=4 |
|---|---|---|---|---|---|
| -.05 | .3275 | | .0062 | .0420 | .1645 |
| .00 | .6562 | .0524 | .2445 | .5465 | .8102 |
| .05 | .8945 | .4583 | .8366 | .9704 | .9967 |
| .10 | .9821 | .8814 | .9937 | .9998 | 1.0000 |

# SECTION 11

# DATA ANALYSIS

## 11.1 The Data

We obtained the following CSA data for analysis:

1. R&F Coal Preparation Plant feed and product samples collected at 30 minute increments during two intensive efforts for 22 and 40 hours, respectively, during April-June, 1980. Each sample was split prior to laboratory analysis.
2. Republic Steel Coal Preparation Plant feed and product samples collected at 30 minute increments during April-June, 1980.
3. Iowa Public Service coal samples daily composites during July-August, 1982.
4. Homer City as-fired coal samples at 15 minute intervals for Units 1 and 2 and at 30 minute intervals for Unit 3 during November 1980-April 1982 (Time 1).

We obtained the following CEM data for analysis:

1. Colorado Public Service hourly averages during June-August, 1982.
2. Possum Point Generating Station hourly averages taken with CSI and DuPont CEM's during March 1982.
3. Possum Point Generating Station measurements taken with CSI and DuPont CEM's at 15 minute intervals during February 1982.
4. Homer City daily averages at Units 1, 2, and 3 during November 1980-April 1982 (Time 1).
5. Homer City three hour averages at Units 1 and 2 during December 1981-February 1982 (Time 2).
6. Homer City daily averages at Unit 3 during December 1981-February 1982 (Time 3).

We obtained the followed Method 6B data for analysis:

1. Homer City three hour averages at Units 1 and 2 during December 1981-February 1982 (Time 2).
2. Homer City daily averages at Units 1, 2, and 3 during December 1981-February 1982 (Time 3).

From these data, we obtained 53 useable data sets: 19 for CSA, 25 for CEM, and 9 for Method 6B. A useable data set had to contain at least 45 (or so) consecutive data points.

## 11.2 A Typical Analysis

One CSA data set we analyzed was the Republic Steel clean coal data set #2 consisting of 226 data points. The data had the following statistical characteristics:

average = 1.3795
variance = 0.0119
first order correlation = 0.5746
second order correlation = 0.4078.

The moment estimators for the auto-regressive and moving-average parameters are (from (6-8) and (6-9)):

auto-regressive = .7097
moving-average = .2046.

More sophisticated estimators are obtainable via a Box-Jenkins Time Series routine. It provided the estimators:

$\widehat{AR}(X)$ = .7998
$\widehat{MA}(X)$ = .3679

with residual error variance, or white noise variance,

WNV(X) = .00758.

Versar implemented a computer routine which computes the coal and measurement error parameters based on these estimates. The results are:

Estimates for the total process X
var(X) = .0115
AR(X) = .7998
MA(X) = .3679

Estimates for the coal process C
var(C) = .0080 = 69.55% of total variance
AR(C) = .7998
MA(C) = -.00019
WNV(C) = .0029

Estimates for the measurement process ME
var (ME) = .0035 = 30.45% of total variance
RSD of error = 1/23

where

RSD of error = standard deviation/estimated mean.

### 11.3 Goodness-of-Fit-Analysis

To determine whether the fitted ARMA(1,1) model adequately fit the data we considered three separate indicators: residual analysis, overfit, and $R^2$.

The residuals are estimated by the difference between the forecast of the next data point at each time and the actual observed data point:

$$\widehat{\text{residual}} = (X_{t+1} - \bar{X}) - \widehat{AR}(X)\,(X_t - \bar{X}) \quad .$$

If the model fits, then these residuals should form an independent sequence of random variables. If they do, then 95% of their correlations should be in the interval $\pm 1.96/\sqrt{n-1}$ and

$$Q = (n)\ (\text{sum of squared correlations})$$

should be small. For our set above, the computer calculated the first twenty correlations for the residuals. We found that 2 of these 20 were not in the interval $\pm .12$ and that Q=21.31. Neither of these tests shows that the ARMA(1,1) model is inadequate (but, on the other hand, neither is very compelling evidence either).

The second method we used was "overfitting" the model. We fit the data with ARMA(1,2) and ARMA(2,1) models to see whether either was significantly better. For the set analyzed above, the white noise variances were .0073 and .00745, not significantly better than .00758.

The third indicator of fit is the overall $R^2$ defined by

$$R^2 = 1 - \frac{\text{residual variance}}{\text{total variance}} = \text{".\% of variance explained by model."}$$

In the above example $R^2 = .748$.

A summary of the model fits and goodness-of-fit criteria is provided in Table 11-1 for our CSA data. Summaries of the models for CEM and Method 6B are provided by Tables 11-2 and 11-3.

## 11.4 Verification of the Coal Model

Assuming that our ARMA(1,1) model fit the observed data, the next issue was whether the coal model described above adequately models the real phenomena. For this, we performed three analyses: positive variances, stationarity, and consistency.

The split of the total variance into two pieces, one due to the coal and the other to the measuring process, is model dependent. It is possible for other ARMA(1,1) models to distribute the total variance into pieces other than those obtained by our model. The fact that we obtained two positive values for the variances is, thus, a first "verification" of the model.

To study stationarity, the second Republic Steel Plant data were split into thirds: the first group contains observations 1 to 88, the second contains observations 89 to 176, and the third contains observations 177 to 264. These three sequences are virtually independent. If the coal properties are stationary, then the mean and variance as well as the time series parameters should be the same for these three series.

Let $\mu_i$ and $\sigma_i^2$ be the population mean and variance for the ith series (i=1,2,3). Estimates for these quantities are provided by the sample means and variances:

| | mean | variance |
|---|---|---|
| Series 1 | 1.4096 | .0098 |
| Series 2 | 1.3958 | .0113 |
| Series 3 | 1.3348 | .0117. |

## TABLE 11-1. SUMMARY OF RESULTS FOR CSA

| Data Set | Average | $\hat{\text{Var}}$(C) | $\hat{\text{Var}}$(ME) | $\text{RSD}^{-1}$(ME) | $R^2$ | AC(Residuals) out of Range[1] | $Q^2$ | Overfit Improvement[3] |
|---|---|---|---|---|---|---|---|---|
| 1. R&F, A, ROM, Even Splits | 2.93 | .0352 | .0109 | 28 | .675 | none | 21.5 | No |
| 2. R&F, A, ROM, Odd Splits | 2.92 | .0358 | .0096 | 29 | .705 | 3 | 29.9 | Yes (Both) |
| 3. R&F, BC, ROM, Even Splits | 3.32 | .0610 | .0668 | 13 | .902 | none | 11.6 | No |
| 4. R&F, BC, ROM, Odd Splits | 3.32 | .0578 | .0594 | 14 | .911 | none | 10.7 | No |
| 5. R&F, A, Clean, Even Splits | 2.53 | .0039 | .00945 | 26 | .797 | none | 11.7 | No |
| 6. R&F, A, Clean, Odd Splits | 2.50 | .0041 | .0087 | 27 | .805 | 1 | 22.2 | Yes (1,2) |
| 7. R&F, BC, Clean, Even Splits | 2.82 | .0557 | .0314 | 16 | .852 | none | 11.2 | No |
| 8. R&F, BC, Clean, Odd Splits | 2.83 | .0591 | .0316 | 16 | .869 | none | 11.9 | No |
| 9. Republic, Clean, Set 1 | 1.42 | .0033 | .0050 | 20 | .849 | 1 | 24.7 | No |
| 10. Republic, Clean, Set 2 | 1.38 | .0080 | .0035 | 23 | .748 | 2 | 21.3 | No |
| 11. Republic, ROM, Set 1 | 2.70 | .0643 | .00375 | 44 | .452 | none | 8.2 | No |
| 12. Republic, ROM, Set 2 | 2.69 | negative split | | | | 1 | 9.8 | Yes (both) |
| 13. Iowa P.S. | 0.54 | negative split | | | | 1 | 24.2 | Yes (1,2) |
| 14. Homer City, Unit 1 (Time 1) | 2.57 | .0107 | .0095 | 26 | .619 | none | 18.4 | No |
| 15. Homer City, Unit 2, Set 1 (Time 1) | 2.49 | negative split | | | | none | 7.3 | No |
| 16. Homer City, Unit 2, Set 2 (Time 1) | 2.76 | .0326 | .0294 | 16 | .583 | none | 8.3 | No |
| 17. Homer City, Unit 3, Set 1 (Time 1) | 2.66 | negative split | | | | none | 11.9 | No |
| 18. Homer City, Unit 3, Set 2 (Time 1) | 2.57 | .0091 | .0147 | 21 | .794 | none | 28.5 | No |
| 19. Homer City, Unit 3, Set 3 (Time 1) | 2.47 | .0282 | .0440 | 12 | .968 | none | 15.0 | No |

---

[1] Expected number out of range is 1, i.e. 5% of 20 is 1.

[2] Significance levels for 10%=26.0, for 5%=28.9, for 1%=34.8.

[3] Yes means white noise variance may be reduced by 10% by ARMA (1,2) or ARMA (2,1).

TABLE 11-2. SUMMARY OF RESULTS FOR CEM

| Data Set | Average | $\widehat{\text{Var}}(C)$ | $\widehat{\text{Var}}(ME)$ | RSD$^{-1}$(ME) | $R^2$ | AC(Residuals) out of Range[1] | $Q^2$ | Overfit Improvement[3] |
|---|---|---|---|---|---|---|---|---|
| 1. Colorado, Set 1 | 0.79 | .0026 | .00026 | 5 | .814 | none | 6.0 | No |
| 2. Colorado, Set 2 | 0.84 | .0056 | .000027 | 162 | .868 | none | 26.2 | No |
| 3. Colorado, Set 3 | 0.70 | .00049 | .00016 | 55 | .831 | 1 | 12.1 | No |
| 4. Colorado, Set 4 | 0.71 | .0070 | .00041 | 35 | .914 | none | 15.3 | No |
| 5. Colorado, Set 5 | 0.72 | .0148 | .000016 | 180 | .935 | none | 18.0 | No |
| 6. Colorado, Set 6 | 0.63 | .0064 | .00015 | 51 | .902 | none | 19.7 | No |
| 7. Colorado, Set 7 | 0.68 | .00078 | .000014 | 182 | .775 | none | 20.0 | No |
| 8. Homer City, Unit 1 (Time 1) | 2.46 | .0285 | .0165 | 19 | .700 | 1 | 13.7 | No |
| 9. Homer City, Unit 2, Set 1 (Time 1) | 2.55 | negative split | | | | none | 10.8 | Yes (1,2) |
| 10. Homer City, Unit 2, Set 2 (Time 1) | 2.57 | negative split | | | | none | 16.8 | No |
| 11. Homer City, Unit 3, Set 1 (Time 1) | 2.65 | negative split | | | | none | 9.0 | No |
| 12. Homer City, Unit 3, Set 2 (Time 1) | 2.50 | .0265 | .0194 | 18 | .878 | 1 | 18.5 | No |
| 13. Possum Point, DuPont, Set 1 | 1.70 | .0375 | .0035 | 29 | .946 | 1 | 19.0 | No |
| 14. Possum Point, DuPont, Set 2 | 1.81 | .2177 | .0027 | 35 | .855 | none | 8.2 | No |
| 15. Possum Point, DuPont, Set 3 | 1.72 | .1113 | .0008 | 60 | .905 | none | 16.6 | No |
| 16. Possum Point, CSI, Set 1 | 1.65 | .0243 | .0030 | 30 | .883 | 2 | 41.6 | No |
| 17. Possum Point, CSI, Set 2 | 1.69 | .2095 | .0091 | 18 | .854 | 1 | 12.6 | No |
| 18. Possum Point, CSI, Set 3 | 1.72 | .1032 | .0008 | 60 | .905 | none | 9.2 | No |
| 19. Homer City, Unit 1, Set 1 (Time 2) | 2.37 | .0198 | .0005 | 106 | .68 | none | 8.5 | No |
| 20. Homer City, Unit 1, Set 2 (Time 2) | 2.47 | negative split | | | | none | 9.5 | No |
| 21. Homer City, Unit 1, Set 3 (Time 2) | 2.44 | .0167 | .0002 | 173 | .61 | none | 13.3 | No |
| 22. Homer City, Unit 2, Set 1 (Time 2) | 2.62 | .0275 | .0016 | 66 | .71 | none | 9.3 | Yes (1,2) |
| 23. Homer City, Unit 2, Set 2 (Time 2) | 2.51 | negative split | | | | 1 | 9.0 | No |
| 24. Homer City, Unit 2, Set 3 (Time 2) | 2.60 | negative split | | | | 2 | 15.4 | No |
| 25. Homer City, Unit 3, (Time 3) | 2.51 | .0331 | .0057 | 33 | .24 | 1 | 12.0 | No |

---

[1] Expected number out of range is 1, i.e. 5% of 20 is 1.

[2] Significance levels for 10%=26.0, for 5%=28.9, for 1%=34.8.

[3] Yes means white noise variance may be reduced by 10% by ARMA (2,1) or ARMA (1,2).

## TABLE 11-3. SUMMARY OF RESULTS FOR METHOD 6B

| | Data Set | Average | $\hat{\text{Var}}$(C) | $\hat{\text{Var}}$(ME) | $\text{RSD}^{-1}$(ME) | $R^2$ | AC(Residuals) out of Range[1] | Q[2] | Overfit Improvement[3] |
|---|---|---|---|---|---|---|---|---|---|
| 1. | Unit 1, Set 1 (Time 2) | 2.59 | .0409 | .0063 | 33 | .81 | 1 | 24.4 | Yes (1,2) |
| 2. | Unit 1, Set 2 (Time 2) | 2.60 | .0305 | .0029 | 48 | .87 | 1 | 9.7 | No |
| 3. | Unit 1, Set 3 (Time 2) | 2.52 | .0269 | .0008 | 89 | .66 | none | 19.5 | No |
| 4. | Unit 2, Set 1 (Time 2) | 2.76 | .0247 | .0025 | 55 | .74 | none | 13.1 | No |
| 5. | Unit 2, Set 2 (Time 2) | 2.45 | .0325 | .0062 | 31 | .55 | none | 11.2 | Yes (Both) |
| 6. | Unit 2, Set 3 (Time 2) | 2.59 | .0249 | .0014 | 69 | .56 | none | 14.0 | No |
| 7. | Unit 1, (Time 3) | 2.59 | .0356 | .0037 | 43 | .25 | 1 | 18.7 | No |
| 8. | Unit 2, (Time 3) | 2.76 | .0236 | .0372 | 14 | .82 | none | 8.8 | No |
| 9. | Unit 3, (Time 3) | 2.54 | Negative Split | | | | none | 13.8 | No |

---

[1] Expected number out of range is 1, i.e. 5% of 20 is 1.

[2] Significance levels for 10%=26.0, for 5%=28.9, for 1%=34.8.

[3] Yes means white noise variance may be reduced by 10% by ARMA (2,1) or ARMA (1,2).

An analysis of variance was performed to test the hypothesis of equal means:

| Source | SS | df | MS | F |
|---|---|---|---|---|
| Between | .97 | 2 | .48 | 44 |
| Among | 2.35 | 261 | .01 | |

Since the F-value is significant at the 99% level, the equality of the means is rejected. Further analysis showed that the mean of Series 3 was significantly smaller than the other two while the other two were not significantly different. Stationarity of the mean apparently does not hold for this long data set. As a consequence, the variance for the total data set includes variations about local means and variation of the local means.

To test the hypothesis of equal variances Bartlett's test was used. Here B=M/C is approximately $x^2$(df=2)

$$M = 1.802$$

$$C = 1.005$$

and so

$$B = 1.793.$$

This value is not significant and so equality of variances can be accepted. Thus the mean but not the variance of the data was changing in time.

The model parameters for the three series are:

| | $\hat{A}R(C)$ | $V\hat{a}r(C)$ | $V\hat{a}r$ME |
|---|---|---|---|
| Series 1 | .5149 | .0075 | .0018 |
| Series 2 | .9366 | .0065 | .0037 |
| Series 3 | .4794 | Negative split. | |
| All data | .7998 | .0080 | .0035. |

These values do not suggest that the time series model describing this coal data set is time independent.

To test the consistency of the modeling procedure, the data were split into three groups: the first group contains the observations ($X_1$, $X_4$, $X_7$,...), the second the observations ($X_2$, $X_5$, $X_8$,...), and the third the observations ($X_3$,$X_6$,$X_9$,...). The parameters describing these series are:

| | Average | Sample variance | $\hat{AR}(C)$ | $\hat{Var}(C)$ | $\hat{Var}ME$ |
|---|---|---|---|---|---|
| Series 1 | 1.37 | .0124 | .6621 | .0082 | .0035 |
| Series 2 | 1.36 | .0104 | .9214 | .0041 | .0063 |
| Series 3 | 1.38 | .0131 | .6047 | .0082 | .0050 |
| All data | 1.38 | .0115 | .7998 | .0080 | .0035 |

Since the three series are highly correlated, standard tests are not applicable. Pairwise testing whether the estimated means were equal produced no significant differences. (Here we used the appropriate standard error of the mean of the differences from time series considerations.)

## 11.5 Comparison of the Models for CSA and CEM

We obtained from Homer City measurements from their CSA and CEM program for the same periods of time (listed above as Time 1). Thus determinations of the underlying coal process (when split off from the measurement errors) was theoretically possible for two different media and comparisons could be made. Our results are presented in Table 11-4. Their usefulness is limited by the following considerations:

1. CSA is performed as-fired rather than the typical as-bunkered.
2. About half the data sets had variance splits with negative measurement errors.

Using a pooled variance estimate and independence (both inappropriate) to compare the averages, only Unit 3, Set 1 have estimates for the means which are consistent. Of the others, CSA is high three times and low once. The estimates for the variance of the coal are not consistent for the two possible comparisons. The estimates for the autocorrelation of the coal are in reasonable agreement.

TABLE 11-4. COMPARISON OF CSA AND CEM FOR HOMER CITY (TIME 1)

| Data Set | Average | $\widehat{\text{Var}}(C)$ | $\widehat{\text{AR}}(C)$ | Sample variance | Number |
|---|---|---|---|---|---|
| Unit 1, CSA | 2.57 | .0107 | .3332 | .0284 | 63 |
| Unit 1, CEM | 2.47 | .0285 | .5886 | .0470 | |
| Unit 2, Set 1, CSA | 2.49 | ** | .2911 | .0268 | 90 |
| Unit 2, Set 1, CEM | 2.55 | ** | .1232 | .0225 | |
| Unit 2, Set 2, CSA | 2.76 | .0326 | .2515 | .0623 | 82 |
| Unit 2, Set 2, CEM | 2.57 | ** | .1137 | .0632 | |
| Unit 3, Set 1, CSA | 2.66 | ** | .2444 | .0132 | 58 |
| Unit 3, Set 1, CEM | 2.65 | ** | .1776 | .0175 | |
| Unit 3, Set 2, CSA | 2.57 | .0091 | .5232 | .0238 | 156 |
| Unit 3, Set 2, CEM | 2.50 | .0265 | .8252 | .0455 | |

## 11.6 Analysis of the R&F Data

Laboratory samples for the R&F Coal Company Preparation Plant were analyzed in duplicate thereby providing us with an opportunity to compare two independent determinations of the underlying coal process. Our results are presented in Table 11-5. Agreement between the split sample streams for all four pairs is virtually perfect.

## 11.7 Measurement Error Analysis

The R&F laboratory samples were analyzed in duplicate (see 11.6). Thus, the difference in the paired measurements is a measure of the laboratory analytic error, an error forming a part of the total measurement error. An analysis of variance was performed on the four paired data sets; results are listed in Table 11-6. Averaging the results in Table 11-5 we have estimates:

| Data Set | Coal Variance | Measurement Error |
|---|---|---|
| A/ROM | .0355 | .0103 |
| BC/ROM | .0594 | .0631 |
| A/Clean | .0040 | .0090 |
| BC/Clean | .0574 | .0315 |

If we now identify the between variance of ANOVA with the laboratory analytical error and the measurement error from above with the sum of sampling, preparation, and analytical errors, we may conclude the following:

| Data Set | Analytical Error | (% of total) | Sampling and Prep Error | (% of total) |
|---|---|---|---|---|
| A/ROM | .0045 | (44%) | .0058 | (56%) |
| BC/ROM | .00265 | ( 4%) | .06045 | (96%) |
| A/Clean | .0044 | (49%) | .0046 | (51%) |
| BC/Clean | .0043 | (14%) | .0272 | (86%) |

Thus we see that three data sets have equal laboratory errors (of about .0044) while the sampling plus preparation errors are extremely variable.

### TABLE 11-5. COMPARISON OF R&F SPLIT DATA SETS

| Data Set | Average | $\widehat{\mathrm{Var}}(C)$ | $\widehat{\mathrm{VAR}}(ME)$ | $\widehat{\mathrm{AR}}(C)$ | Number |
|---|---|---|---|---|---|
| A/ROM/Even | 2.93 | .0352 | .0109 | .6432 | 44 |
| A/ROM/Odd | 2.92 | .0358 | .0096 | .6989 | |
| BC/ROM/Even | 3.32 | .0610 | .0668 | .8337 | 62 |
| BC/ROM/Odd | 3.32 | .0578 | .0594 | .8555 | |
| A/Clean/Even | 2.53 | .0039 | .0095 | .3727 | 41 |
| A/Clean/Odd | 2.50 | .0041 | .0087 | .4607 | |
| BC/Clean/Even | 2.82 | .0557 | .0314 | .8130 | 82 |
| BC/Clean/Odd | 2.83 | .0591 | .0316 | .8385 | |

## TABLE 11-6. ANOVA FOR PAIRED R&F DATA

1. A/ROM Data

| Source | SS | df | MS | Variance |
|---|---|---|---|---|
| Among | 4.34 | 43 | .10105 | .04828 |
| Between | 0.20 | 44 | .00450 | .00450 |

2. BC/ROM Data

| Source | SS | df | MS | Variance |
|---|---|---|---|---|
| Among | 15.38 | 61 | .25208 | .12472 |
| Between | 0.16 | 62 | .00265 | .00265 |

3. A/Clean Data

| Source | SS | df | MS | Variance |
|---|---|---|---|---|
| Among | 1.20 | 42 | .02847 | .01206 |
| Between | 0.19 | 43 | .00436 | .00436 |

4. BC/Clean Data

| Source | SS | df | MS | Variance |
|---|---|---|---|---|
| Among | 13.80 | 79 | .17468 | .08518 |
| Between | 0.35 | 80 | .00432 | .00432 |

## 11.8 Conclusions

The ARMA (1,1) model fits exceptionally well, especially as it accounts only for auto-correlation between measurements, leaving out any dependence on weather; season of year; temperature, specifications, and efficiency of the boiler; load; blending practices; spot coal versus contract coal supplies; etc. It does about as well for data obtained via CSA as it does for CEM and Method 6B generated data. There is little indication of non-goodness-of-fit (as measured by our analyses of residuals) nor that an additional parameter would explain significantly greater percentage of the variance. All in all, the model presented above provides a very satisfactory, first generation, universally applicable model.

# SECTION 12

# CORRELATION OF CSA DATA TO $SO_2$ EMISSIONS

The ultimate value of a CSA program will be based on the accuracy of predicted $SO_2$ gaseous emissions for coal feedstock data. The behavior of the sulfur in coal must be considered from the sampling point to the emission point. If the parameters that affect the mass balance of sulfur in the system can be identified, then an estimate can be made of the sulfur loss.

Sulfur loss in the pulverizers and in the boiler has already been discussed (Section 5). Parameters affecting sulfur loss at these sites probably include: amount of pyritic sulfur in the coal, alkaline content and composition of ash, fuel additives, boiler size, boiler firing type, and boiler firing rate (percent excess air).

A major factor that must be accounted for in the simulation of sulfur loss is the $SO_2$ removal efficiency of pollution control devices. Systems such as flue gas desulfurization units may trap a high proportion of $SO_2$. Particulate collection devices such as electrostatic precipitators and mechanical dust collectors will not show high $SO_2$ removal but may contribute by removing $SO_2$ or $SO_3$ adsorbed to fly ash particles.

Data sets which include CSA data, emissions data, and boiler operating parameters are virtually non-existent. For this reason, no causal analytic model could be developed. In lieu of an explanatory model, we developed the stochastic model described in Section 8.

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## Table of Conversion Factors

| Multiply English Unit | by | To Obtain SI Unit |
|---|---|---|
| pound (lb) | 453.59 | gram |
| ton (2000 lb) | 0.907 | megagram (Mg) = metric ton |
| inch (in.) | 0.0254 | meter (m) |
| foot (ft) | 3.048 | meter (m) |
| British Thermal Unit (Btu) | 1054.88 | Joule (J) |
| Btu/lb | 2.326 | J/g |
| lb/$10^6$Btu | 429.907 | ng/J |
| gallon/minute (gpm) | 0.06309 | liter/second (l/s) |

Tyler Screen Size Mesh Openings

| Mesh Size | in. | mm |
|---|---|---|
| 14 | 0.0469 | 1.18 |
| 24 | 0.0234 | 0.60 |
| 48 | 0.0117 | 0.30 |
| 100 | 0.0059 | 0.15 |
| 200 | 0.0029 | 0.075 |
| 270 | 0.0021 | 0.053 |
| 325 | 0.0017 | 0.045 |

# Appendix A

# DATA SETS

```
  1.      DATA SET A, R & F COAL CO., ROM CSA DATA 30 MIN EVEN SPLITS
  2.               3.2636242
  3.               2.9491444
  4.               3.0006313
  5.               3.0714207
  6.               3.0636864
  7.               3.1259794
  8.               3.1471443
  9.               3.4042101
 10.               2.8887711
 11.               2.8985510
 12.               2.7754669
 13.               2.8903551
 14.               2.6712751
 15.               2.5651798
 16.               2.4531479
 17.               2.7055359
 18.               2.8029585
 19.               2.9278555
 20.               2.9626493
 21.               3.0939808
 22.               2.9809713
 23.               3.2199974
 24.               3.0945129
 25.               2.5018396
 26.               2.8735628
 27.               2.9018784
 28.               2.6149960
 29.               2.7186508
 30.               2.5402193
 31.               2.7379808
 32.               2.6430311
 33.               2.8498774
 34.               2.8112440
 35.               2.9598303
 36.               3.3862429
 37.               3.1615553
 38.               3.1236668
 39.               3.1700583
 40.               3.0926743
 41.               3.1203718
 42.               2.9746094
 43.               2.7716064
 44.               2.8477650
 45.               2.8762465
 1.      DATA SET A, R & F COAL CO., ROM CSA DATA 30 MIN ODD SPLITS
 2.                 3.3375568
 3.                 2.9283037
 4.                 3.0532732
 5.                 3.1240139
 6.                 3.1161470
 7.                 3.1782532
 8.                 3.2409325
 9.                 3.3308887
10.                 2.9836578
11.                 3.0211811
12.                 2.7754669
13.                 2.7646875
14.                 2.5979462
15.                 2.6387720
16.                 2.3794479
17.                 2.9152679
18.                 2.8029585
19.                 2.7277508
20.                 3.1213617
21.                 3.0728607
22.                 2.9069757
23.                 3.0823002
24.                 2.9791250
25.                 2.5438871
26.                 2.7795191
27.                 2.7766171
28.                 2.5834904
29.                 2.6238136
30.                 2.6354780
31.                 2.6106329
32.                 2.6854887
33.                 2.6591797
34.                 2.8112440
35.                 3.0549679
36.                 3.2698412
37.                 3.0877857
38.                 3.1663113
39.                 3.0958433
40.                 3.0926743
41.                 3.1203718
42.                 2.9108667
43.                 2.8456564
44.                 2.8265924
45.                 3.0036077
```

```
                                                                     54.        3.2637482
  1.      DATA SETS B-C, R & F COAL CO.,ROM CSA DATA 30 MIN EVEN SPL55S        2.8709631
  2.       3.5984430                                                 56.        3.1086454
  3.       3.6167717                                                 57.        2.8777733
  4.       3.4803028                                                 58.        3.0691071
  5.       3.4511957                                                 59.        2.6014891
  6.       3.6591845                                                 60.        3.3228006
  7.       3.3788099                                                 61.        3.0453625
  8.       3.2048550                                                 62.        3.1944151
  9.       3.1134167                                                 63.        2.5050411
 10.       3.1593704                                                 64.        3.4937725
 11.       2.4768124
 12.       3.4567366
 13.       2.9732304
 14.       3.1217041
 15.       3.2105761
 16.       3.2121267
 17.       3.5278006
 18.       3.7807579
 19.       3.9327621
 20.       3.2757339
 21.       3.6564798
 22.       3.9331779
 23.       3.6228809
 24.       4.0679760
 25.       4.0063925
 26.       3.5063562
 27.       3.5292873
 28.       3.4511776
 29.       3.4767857
 30.       3.1717396
 31.       3.7640095
 32.       3.2189512
 33.       3.2679739
 34.       3.2213764
 35.       3.0562601
 36.       3.4713373
 37.       3.4928026
 38.       3.5778360
 39.       3.5445185
 40.       3.6887846
 41.       4.2415371
 42.       6.0599566
 43.       3.2423201
 44.       2.9302073
 45.       3.3033342
 46.       2.7899990
 47.       3.0067987
 48.       3.2017069
 49.       3.0185509
 50.       3.6507931
 51.       3.1292801
 52.       3.0417023
 53.       2.7597914
 1.     DATA SETS B-C, R & F COAL CO.,ROM CSA DATA 30 MIN ODD SPLI55.        2.9025135
 2.      3.5119944                                                 56.        3.0772438
 3.      3.6061020                                                 57.        2.9414883
 4.      3.4269238                                                 58.        3.1749382
 5.      3.4088497                                                 59.        2.5490398
 6.      3.5315380                                                 60.        3.3024149
 7.      3.3361750                                                 61.        3.1511040
 8.      3.3219757                                                 62.        3.3001900
 9.      3.1663656                                                 63.        2.4519691
10.      3.2132835                                                 64.        3.4937725
11.      2.5189714
12.      3.4247303
13.      3.0049725
14.      3.1111574
15.      3.1790991
16.      3.3817444
17.      3.6225767
18.      3.7490759
19.      3.8799019
20.      3.3393393
21.      3.6564798
22.      3.8591661
23.      3.6440668
24.      4.0148687
25.      4.0596695
26.      3.5487289
27.      3.5082169
28.      3.5248318
29.      3.5085859
30.      3.1399164
31.      3.6688519
32.      3.1233387
33.      3.3839340
34.      3.1474428
35.      3.0985613
36.      3.3970270
37.      3.4398813
38.      3.6200523
39.      3.4914560
40.      3.6993847
41.      4.0848303
42.      6.0599566
43.      3.3063135
44.      2.8449650
45.      3.2398090
46.      2.9187679
47.      3.0599232
48.      3.1696901
49.      2.9451838
50.      3.4814806
51.      3.0133801
52.      3.0417023
53.      2.7810202
54.      3.2319593
```

```
  1.      DATA SET A, R & F COAL CO., CLEAN COAL CSA DATA 30 MIN EVEN SPLITS
  2.              2.9494543
  3.              2.7327814
  4.              2.4894886
  5.              2.4883871
  6.              2.7424746
  7.              2.5550652
  8.              2.5086851
  9.              2.4623060
 10.              2.4363585
 11.              2.4097643
 12.              2.4958944
 13.              2.2255030
 14.              2.3482122
 15.              2.5635567
 16.              2.4769764
 17.              2.5498428
 18.              2.6274300
 19.              2.5851192
 20.              2.5562801
 21.              2.5874853
 22.              2.5567875
 23.              2.4567690
 24.              2.6081419
 25.              2.3582897
 26.              2.6174698
 27.              2.4418354
 28.              2.3095407
 29.              2.3488512
 30.              2.6343403
 31.              2.5571146
 32.              2.5866499
 33.              2.6161861
 34.              2.4949350
 35.              2.8107634
 36.              2.4770737
 37.              2.5751972
 38.              2.6701841
 39.              2.5711851
 40.              2.6575212
 41.              2.4909897
 42.              2.3613625
 43.              2.4876661
 44.              2.5366268
 1.      DATA SET A, R & F COAL CO., CLEAN COAL CSA DATA 30 MIN ODD SPLITS
 2.                 2.7536402
 3.                 2.6336088
 4.                 2.4120378
 5.                 2.4330893
 6.                 2.5863981
 7.                 2.3788538
 8.                 2.4476976
 9.                 2.5165415
10.                 2.3271055
11.                 2.3676729
12.                 2.3973722
13.                 2.3415251
14.                 2.4222546
15.                 2.6912022
16.                 2.4981470
17.                 2.4029379
18.                 2.6799784
19.                 2.4800329
20.                 2.5878391
21.                 2.6405087
22.                 2.5779181
23.                 2.4989452
24.                 2.5657330
25.                 2.3159885
26.                 2.4913273
27.                 2.2951155
28.                 2.3826933
29.                 2.3804502
30.                 2.4454231
31.                 2.3894358
32.                 2.4921751
33.                 2.5949154
34.                 2.5055971
35.                 2.8543406
36.                 2.5192366
37.                 2.5540895
38.                 2.5963058
39.                 2.5180616
40.                 2.5610000
41.                 2.4909897
42.                 2.4575272
43.                 2.5401487
44.                 2.5694294
```

```
                                                                  54.            2.8466377
  1.      DATA SETS B AND C, R & F COAL CO., CLEAN COAL CSA DATA EVEN SPLITS    3.1897087
  2.               2.6281767                                      56.            3.0694838
  3.               2.8796368                                      57.            3.5808592
  4.               3.0981989                                      58.            3.3193712
  5.               2.6105251                                      59.            3.5960684
  6.               2.6932821                                      60.            2.8778477
  7.               2.5126152                                      61.            2.8266268
  8.               2.7850752                                      62.            2.7754059
  9.               2.7489243                                      63.            2.6920614
 10.               2.3894739                                      64.            2.4325752
 11.               2.8799658                                      65.            2.5489779
 12.               3.1952906                                      66.            2.5991793
 13.               3.1600075                                      67.            2.7570238
 14.               3.2551250                                      68.            2.5164452
 15.               2.8535185                                      69.            2.5230761
 16.               3.2397404                                      70.            2.5525694
 17.               2.7391434                                      71.            2.6568031
 18.               2.8335800                                      72.            2.5119858
 19.               3.0373335                                      73.            2.4277935
 20.               2.4832354                                      74.            2.2336407
 21.               2.7137461                                      75.            2.4046431
 22.               2.6840010                                      76.            2.6014891
 23.               2.4405565                                      77.            2.6631422
 24.               2.4433718                                      78.            2.6573572
 25.               2.7001562                                      79.            2.6302099
 26.               2.4550772                                      80.            2.7049522
 27.               2.5952101                                      81.            2.6461658
 28.               2.8835917
 29.               2.7282267
 30.               2.8376961
 31.               2.6542168
 32.               2.5437851
 33.               2.8164616
 34.               3.2341919
 35.               3.0092344
 36.               3.0912848
 37.               2.9959421
 38.               3.1226568
 39.               3.2493715
 40.               3.0096769
 41.               3.3272133
 42.               2.8994570
 43.               3.0281029
 44.               3.0381584
 45.               3.2040644
 46.               3.0598335
 47.               2.4234819
 48.               2.5597973
 49.               3.2190199
 50.               2.6332293
 51.               2.7312031
 52.               2.7684565
 53.               3.3732424
 1.       DATA SETS B AND C, R & F COAL CO., CLEAN COAL CSA DATA O55.SPLITS      54.                  2.7731094
 2.                 2.7251177                                                                        3.2001677
 3.                 2.7502155                                                     56.                  3.1746035
 4.                 3.0874777                                                     57.                  3.6442375
 5.                 2.6842098                                                     58.                  3.3193712
 6.                 2.7355299                                                     59.                  3.7215137
 7.                 2.6808243                                                     60.                  2.7833204
 8.                 2.7640562                                                     61.                  2.8540850
 9.                 2.7489243                                                     62.                  2.9248495
10.                 2.4842100                                                     63.                  2.7665462
11.                 2.9220085                                                     64.                  2.6335268
12.                 3.0166063                                                     65.                  2.4225826
13.                 3.2234201                                                     66.                  2.5676088
14.                 3.3291054                                                     67.                  2.6623163
15.                 2.9169302                                                     68.                  2.5373287
16.                 3.3261328                                                     69.                  2.5230761
17.                 2.7391434                                                     70.                  2.7437296
18.                 2.7912874                                                     71.                  2.4901018
19.                 3.0795183                                                     72.                  2.4390240
20.                 2.5565796                                                     73.                  2.5010462
21.                 2.7032681                                                     74.                  2.4328852
22.                 2.6210937                                                     75.                  2.3528194
23.                 2.5767250                                                     76.                  2.5490398
24.                 2.3175325                                                     77.                  2.7139654
25.                 2.6268969                                                     78.                  2.6365147
26.                 2.6013365                                                     79.                  2.5574379
27.                 2.5952101                                                     80.                  2.5384922
28.                 2.8623104                                                     81.                  2.6461658
29.                 2.7072401
30.                 2.7434549
31.                 2.8220720
32.                 2.6167641
33.                 2.9203901
34.                 3.2133942
35.                 2.8743382
36.                 3.0082979
37.                 3.1103706
38.                 3.1308041
39.                 3.1512375
40.                 3.1479311
41.                 3.4304714
42.                 2.9933243
43.                 2.9451408
44.                 3.1314802
45.                 3.2040644
46.                 2.9969597
47.                 2.4966049
48.                 2.4758701
49.                 3.1769409
50.                 2.6854744
51.                 2.6152048
52.                 2.7160234
53.                 3.3420086
```

```
 1.      REPUBLIC STEEL CLEAN CSA DATA 30 MIN 1ST SET
 2.                  1.4240332
 3.                  1.4240332
 4.                  1.5250778
 5.                  1.6002569
 6.                  1.4343386
 7.                  1.4130888
 8.                  1.5550747
 9.                  1.5442762
10.                  1.4987135
11.                  1.4539757
12.                  1.4799604
13.                  1.4259472
14.                  1.5041599
15.                  1.4934921
16.                  1.3051443
17.                  1.4130087
18.                  1.4480124
19.                  1.4262381
20.                  1.3574648
21.                  1.3353930
22.                  1.3633928
23.                  1.4063339
24.                  1.4008026
25.                  1.3899441
26.                  1.3525114
27.                  1.4061832
28.                  1.4299631
29.                  1.4732952
30.                  1.4188356
31.                  1.4858665
32.                  1.5188789
33.                  1.4440050
34.                  1.3545723
35.                  1.4629393
36.                  1.2574596
37.                  1.4599314
38.                  1.3311682
39.                  1.4826832
40.                  1.3551903
41.                  1.5191259
42.                  1.4674368
43.                  1.3924589
44.                  1.4002838
45.                  1.4002838
46.                  1.4236403
47.                  1.4236403
48.                  1.5288467
49.                  1.4507303
50.                  1.5074816
51.                  1.5294895
52.                  1.4004364
53.                  1.4770241
```

```
54.                  1.3442039
55.                  1.3772583
56.                  1.4347820
57.                  1.5108690
58.                  1.5233355
59.                  1.4801207
60.                  1.5614967
61.                  1.5614967
62.                  1.4462357
63.                  1.5124741
64.                  1.3344898
65.                  1.3561888
66.                  1.4585629
67.                  1.3149166
68.                  1.5381279
69.                  1.4298086
70.                  1.4322090
71.                  1.4322090
72.                  1.3613710
73.                  1.2396231
74.                  1.2292433
75.                  1.2184601
76.                  1.2998457
77.                  1.2667980
78.                  1.2937584
79.                  1.3046303
80.                  1.3966475
81.                  1.5148249
82.                  1.1976042
83.                  1.1976042
84.                  1.5109291
85.                  1.5645084
86.                  1.3262596
87.                  1.3050385
88.                  1.5366545
89.                  1.5366545
90.                  1.3867817
91.                  1.3109417
```

```
                                                                 54.              1.3269253
  1.    REPUBLIC STEEL CLEAN CSA DATA 30 MIN 2ND SET             55.              1.3269253
  2.            1.6142635                                        56.              1.4048262
  3.            1.4454517                                        57.              1.4798927
  4.            1.5036783                                        58.              1.4850969
  5.            1.3330488                                        59.              1.4331703
  6.            1.3674650                                        60.              1.2752171
  7.            1.3018255                                        61.              1.2438583
  8.            1.3944006                                        62.              1.5537796
  9.            1.4694834                                        63.              1.4443588
 10.            1.3457670                                        64.              1.3221912
 11.            1.4305391                                        65.              1.3539238
 12.            1.3674450                                        66.              1.5136223
 13.            1.3123064                                        67.              1.5388498
 14.            1.3618460                                        68.              1.3614120
 15.            1.3299284                                        69.              1.3933201
 16.            1.3999357                                        70.              1.3608332
 17.            1.4105415                                        71.              1.3183069
 18.            1.5112810                                        72.              1.4302874
 19.            1.4048519                                        73.              1.4739599
 20.            1.3330507                                        74.              1.3928757
 21.            1.3224707                                        75.              1.5098343
 22.            1.3974161                                        76.              1.1308727
 23.            1.3974161                                        77.              1.3285017
 24.            1.4066629                                        78.              1.3912849
 25.            1.3749332                                        79.              1.3589296
 26.            1.3957481                                        80.              1.5302792
 27.            1.3742743                                        81.              1.4217491
 28.            1.2741547                                        82.              1.5005808
 29.            1.2741547                                        83.              1.5217152
 30.            1.5278673                                        84.              1.6596613
 31.            1.4740686                                        85.              1.7243233
 32.            1.4726133                                        86.              1.5075369
 33.            1.3984528                                        87.              1.5874825
 34.            1.5002699                                        88.              1.6159372
 35.            1.4355097                                        89.              1.5938005
 36.            1.3632965                                        90.              1.4967766
 37.            1.3313446                                        91.              1.4749260
 38.            1.2826033                                        92.              1.4187489
 39.            1.3688297                                        93.              1.5169706
 40.            1.2346973                                        94.              1.6632938
 41.            1.2870140                                        95.              1.6632938
 42.            1.4661808                                        96.              1.4486380
 43.            1.3912668                                        97.              1.5035114
 44.            1.3212566                                        98.              1.6334133
 45.            1.4704313                                        99.              1.5785999
 46.            1.4055796                                       100.              1.5658922
 47.            1.3626614                                       101.              1.6344719
 48.            1.3839712                                       102.              1.4789658
 49.            1.4054289                                       103.              1.5008755
 50.            1.3908062                                       104.              1.5421629
 51.            1.4438896                                       105.              1.5531006
 52.            1.3117523
 53.            1.3434887
```

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106.        1.4288826          159.        1.3139763
107.        1.5161428          160.        1.4885416
108.        1.3938408          161.        1.4885416
109.        1.3722315          162.        1.4006786
110.        1.3399229          163.        1.4431229
111.        1.3399229          164.        1.3178701
112.        1.5710382          165.        1.2335262
113.        1.4116564          166.        1.3566923
114.        1.4687605          167.        1.3994226
115.        1.4009705          168.        1.3442869
116.        1.3453903          169.        1.3549557
117.        1.3895016          170.        1.4552784
118.        1.4669657          171.        1.4871454
119.        1.2849331          172.        1.4171543
120.        1.3751554          173.        1.3958445
121.        1.4092503          174.        1.3899117
122.        1.3788576          175.        1.3793831
123.        1.3898010          176.        1.4005604
124.        1.4098358          177.        1.4436541
125.        1.2568293          178.        1.2538519
126.        1.5313063          179.        1.3282328
127.        1.4178762          180.        1.2670250
128.        1.4319544          181.        1.2775841
129.        1.4319544          182.        1.2561731
130.        1.3663902          183.        1.1702805
131.        1.3884296          184.        1.3913965
132.        1.3927269          185.        1.3382893
133.        1.4258862          186.        1.2996769
134.        1.3629150          187.        1.2781944
135.        1.4368715          188.        1.3016996
136.        1.2619295          189.        1.2492113
137.        1.2619295          190.        1.3662395
138.        1.3041639          191.        1.3111496
139.        1.1989899          192.        1.2950382
140.        1.2915716          193.        1.3785896
141.        1.2269936          194.        1.3592014
142.        1.3595161          195.        1.4578533
143.        1.3595161          196.        1.5208311
144.        1.2445011          197.        1.6386414
145.        1.2445011          198.        1.3986006
146.        1.3236542          199.        1.5045557
147.        1.3770275          200.        1.4833918
148.        1.2893677          201.        1.4833918
149.        1.2576609          202.        1.5604095
150.        1.2802725          203.        1.3597860
151.        1.2375965          204.        1.3931723
152.        1.2219954          205.        1.5101557
153.        1.2648726          206.        1.2651548
154.        1.1900959          207.        1.3600416
155.        1.2644768          208.        1.2451200
156.        1.3606882          209.        1.2662230
157.        1.3713188          210.        1.4359188
158.        1.3139763          211.        1.4680662
                               212.        1.4030199
```

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                                                    266.          1.3331890
213.          1.4030199                             267.          1.2901831
214.          1.3589792
215.          1.3798866
216.          1.3004713
217.          1.2794962
218.          1.1429176
219.          1.2070065
220.          1.0938148
221.          1.1148510
222.          1.3121099
223.          1.2905998
224.          1.2218437
225.          1.2962170
226.          1.2821856
227.          1.3032045
228.          1.3176212
229.          1.3069096
230.          1.1816368
231.          1.1816368
232.          1.2539511
233.          1.2855635
234.          1.3196478
235.          1.3824873
236.          1.1668234
237.          1.1983604
238.          1.4060316
239.          1.4382305
240.          1.3069773
241.          1.3393812
242.          1.2931490
243.          1.2931490
244.          1.5208311
245.          1.4779902
246.          1.4778318
247.          1.4778318
248.          1.2664795
249.          1.2457170
250.          1.3465471
251.          1.3788633
252.          1.1188517
253.          1.1821823
254.          1.5153227
255.          1.5153227
256.          1.4031601
257.          1.3589649
258.          1.2765055
259.          1.2026587
260.          1.3235130
261.          1.2914025
262.          1.3929377
263.          1.3929377
264.          1.4011641
265.          1.3580513
```

```
 1.     REPUBLIC STEEL ROM CSA DATA 30 MIN 1st SET            54.            2.7402983
 2.                 2.1851568                                 55.            2.7402983
 3.                 2.1534882                                 56.            2.1904755
 4.                 3.3053503                                 57.            2.2328033
 5.                 3.2738714                                 58.            2.9969597
 6.                 2.6835117                                 59.            2.9969597
 7.                 2.7043953                                 60.            3.3427629
 8.                 2.3379049                                 61.            3.1646233
 9.                 2.3171225                                 62.            2.5532351
10.                 3.0875139                                 63.            2.6255941
11.                 2.9310522
12.                 2.3263083
13.                 2.3993320
14.                 2.3663721
15.                 2.2833414
16.                 2.5664711
17.                 2.5562048
18.                 2.1552162
19.                 2.2173862
20.                 2.3168039
21.                 2.3587370
22.                 2.8982477
23.                 2.7731438
24.                 2.8827868
25.                 2.8405485
26.                 2.4952612
27.                 2.6216040
28.                 2.3246069
29.                 2.4397898
30.                 2.7559891
31.                 2.7241879
32.                 2.8867273
33.                 2.9285631
34.                 2.6887932
35.                 2.6887932
36.                 2.9680357
37.                 3.1133242
38.                 2.5433769
39.                 2.8559103
40.                 2.4953203
41.                 2.5369091
42.                 2.9830570
43.                 3.0038452
44.                 2.5023556
45.                 2.5861158
46.                 2.6447830
47.                 2.6447830
48.                 2.6259832
49.                 2.4239836
50.                 3.3000841
51.                 3.3213072
52.                 3.1187649
53.                 3.1187649
```

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 1.     REPUBLIC STEEL ROM CSA DATA 30 MIN 2ND SET    54.        2.9175472
 2.            3.0347729                              55.        2.8858337
 3.            2.8977871                              56.        3.1220722
 4.            2.6588984                              57.        3.1949205
 5.            2.8177958                              58.        2.9730301
 6.            3.0149155                              59.        3.0261202
 7.            2.9514437                              60.        2.6216831
 8.            3.0510244                              61.        2.7574682
 9.            3.0299835                              62.        2.8993139
10.            2.8098812                              63.        2.7938843
11.            2.8735018                              64.        2.5238237
12.            2.8073158                              65.        2.6180744
13.            2.9987230                              66.        2.9177990
14.            2.8434525                              67.        3.0537539
15.            2.8434525
16.            2.8318758
17.            2.8318758
18.            2.8451881
19.            2.9393301
20.            2.4772654
21.            2.3727398
22.            2.9690590
23.            2.8232107
24.            3.0426588
25.            3.0112915
26.            2.4461937
27.            2.5721779
28.            2.4239883
29.            2.4448853
30.            2.7870560
31.            2.7870560
32.            3.1581125
33.            3.1685019
34.            2.5463686
35.            2.6511574
36.            2.9839725
37.            2.8785315
38.            3.2160053
39.            3.0169697
40.            2.2834396
41.            2.4405565
42.            2.9160585
43.            2.9160585
44.            2.8895655
45.            2.9315958
46.            2.3043385
47.            2.3043385
48.            2.9319696
49.            2.7337227
50.            3.1944876
51.            3.1109705
52.            2.6795950
53.            2.5544777
```

```
 1.      IOWA PUBLIC SERVICE CSA DATA
 2.                0.3780000
 3.                0.5530000
 4.                0.5450000
 5.                0.4080000
 6.                0.4310000
 7.                0.5080000
 8.                0.4970000
 9.                0.4710000
10.                0.4200000
11.                0.3970000
12.                0.4710000
13.                0.4410000
14.                0.4390000
15.                0.4830000
16.                0.5370000
17.                0.4770000
18.                0.4650000
19.                0.4840000
20.                0.5090000
21.                0.4290000
22.                0.3880000
23.                0.3750000
24.                0.3210000
25.                0.3810000
26.                0.3780000
27.                0.3300000
28.                0.3360000
29.                0.3590000
30.                0.3920000
31.                0.4970000
32.                0.5840000
33.                0.5480000
34.                0.5270000
35.                0.5050000
36.                0.5390000
37.                0.6810000
38.                0.5930000
39.                0.5560000
40.                0.5320000
41.                0.5380000
42.                0.6250000
43.                0.6730000
44.                0.7120000
45.                0.7110000
46.                0.5320000
47.                0.6300000
48.                0.6780000
49.                0.7890000
50.                0.6700000
51.                0.6560000
52.                0.7450000
53.                0.6340000
```

```
54.                0.6480000
55.                0.8600000
56.                0.9280000
57.                0.6250000
58.                0.5830000
59.                0.6190000
60.                0.5680000
61.                0.6340000
62.                0.6390000
63.                0.6390000
64.                0.5110000
65.                0.4750000
66.                0.4800000
67.                0.6010000
68.                0.6330000
69.                0.7830000
70.                0.7120000
71.                0.6200000
72.                0.5800000
73.                0.6520000
74.                0.7300000
75.                0.6970000
76.                0.5790000
77.                0.5400000
78.                0.6230000
79.                0.5600000
80.                0.5360000
81.                0.4890000
82.                0.4610000
83.                0.4280000
84.                0.4760000
85.                0.4710000
86.                0.4630000
87.                0.4940000
88.                0.4650000
89.                0.4120000
90.                0.4700000
91.                0.4230000
92.                0.4400000
93.                0.4040000
```

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                                                                   54.                2.5400000
  1.     HOMER CITY PLANT UNIT 1 CSA AND CEM DATA DAILY OVERLAPPING55.                2.5600004
  2.              3.2399998                                        56.                2.3999996
  3.              2.8599997                                        57.                2.5100002
  4.              2.3999996                                        58.                2.3800001
  5.              2.7700005                                        59.                2.2900000
  6.              2.4799995                                        60.                2.6400003
  7.              2.8199997                                        61.                2.6400003
  8.              2.5799999                                        62.                2.4399996
  9.              2.6499996                                        63.                2.2600002
 10.              2.4899998                                        64.                2.4200001
 11.              2.5400000
 12.              2.5699997
 13.              2.4399996
 14.              2.7200003
 15.              2.5699997
 16.              2.8000002
 17.              2.7299995
 18.              2.5000000
 19.              2.4799995
 20.              2.3900003
 21.              2.3699999
 22.              2.3800001
 23.              2.5200005
 24.              2.7600002
 25.              2.6400003
 26.              2.6099997
 27.              2.5500002
 28.              2.4300003
 29.              2.6199999
 30.              2.6599998
 31.              2.7700005
 32.              2.8000002
 33.              2.7200003
 34.              2.5699997
 35.              2.5900002
 36.              2.7100000
 37.              2.1800003
 38.              2.3699999
 39.              2.6300001
 40.              2.5000000
 41.              2.5900002
 42.              2.4600000
 43.              2.4200001
 44.              2.4099998
 45.              2.5100002
 46.              2.5799999
 47.              2.6499996
 48.              2.5500002
 49.              2.4300003
 50.              2.4899998
 51.              2.6199999
 52.              2.7900000
 53.              2.6700001
```

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                                                                    54.             2.6199999
  1.     HOMER CITY PLANT UNIT 2 CSA AND CEM DATA DAILY OVERLAPPING55.             2.1300001
  2.               2.5200005                                        56.             2.3900003
  3.               2.4099998                                        57.             2.5600004
  4.               2.3699999                                        58.             2.6000004
  5.               2.4200001                                        59.             2.6999998
  6.               2.0900002                                        60.             2.6099997
  7.               2.1599998                                        61.             2.4600000
  8.               2.1999998                                        62.             2.6400003
  9.               2.2900000                                        63.             2.6199999
 10.               2.3800001                                        64.             2.5000000
 11.               2.4099998                                        65.             2.5799999
 12.               3.0699997                                        66.             2.5000000
 13.               2.8000002                                        67.             2.3599997
 14.               2.6099997                                        68.             2.4399996
 15.               2.4300003                                        69.             2.4499998
 16.               2.3699999                                        70.             2.3599997
 17.               2.4899998                                        71.             2.4200001
 18.               2.6400003                                        72.             2.4200001
 19.               2.8199997                                        73.             2.3000002
 20.               2.6499996                                        74.             2.4700003
 21.               2.5400000                                        75.             2.4600000
 22.               2.6199999                                        76.             2.6999998
 23.               2.7399998                                        77.             2.6000004
 24.               2.8299999                                        78.             2.6199999
 25.               2.6999998                                        79.             2.5000000
 26.               2.5799999                                        80.             2.4300003
 27.               2.4600000                                        81.             2.4499998
 28.               2.3999996                                        82.             2.3100004
 29.               2.5100002                                        83.             2.4499998
 30.               2.5000000                                        84.             2.6899996
 31.               2.6199999                                        85.             2.4899998
 32.               2.4700003                                        86.             2.1400003
 33.               2.4799995                                        87.             2.0600004
 34.               2.4600000                                        88.             2.4499998
 35.               2.2700005                                        89.             2.5299997
 36.               2.4499998                                        90.             2.3599997
 37.               2.3299999                                        91.             2.5200005
 38.               2.4099998
 39.               2.3800001
 40.               2.6499996
 41.               2.5000000
 42.               2.4799995
 43.               2.5100002
 44.               2.6700001
 45.               2.5600004
 46.               2.3900003
 47.               2.4799995
 48.               2.3599997
 49.               2.3500004
 50.               2.5900002
 51.               2.5900002
 52.               2.4499998
 53.               2.5699997
```

```
                                                                      54.                 2.5100002
  1.      HOMER CITY PLANT UNIT 2 CSA AND CEM DATA DAILY 2ND OVERLAP55NG                  2.6300001
  2.                2.6300001                                         56.                 2.6999998
  3.                2.2500000                                         57.                 2.6300001
  4.                2.8299999                                         58.                 2.6199999
  5.                2.8900003                                         59.                 2.5799999
  6.                2.5200005                                         60.                 2.5699997
  7.                2.8000002                                         61.                 2.4300003
  8.                2.8400002                                         62.                 2.5600004
  9.                2.8400002                                         63.                 2.9200001
 10.                2.8400002                                         64.                 2.8500004
 11.                2.7700005                                         65.                 2.6199999
 12.                2.9700003                                         66.                 2.5900002
 13.                2.7799997                                         67.                 2.8199997
 14.                2.9399996                                         68.                 2.8000002
 15.                3.1099997                                         69.                 2.4600000
 16.                3.8000002                                         70.                 2.8800001
 17.                3.1300001                                         71.                 2.7200003
 18.                2.7399998                                         72.                 2.9099998
 19.                2.7900000                                         73.                 2.9499998
 20.                2.5900002                                         74.                 2.5100002
 21.                2.7100000                                         75.                 2.7500000
 22.                3.2700005                                         76.                 2.7500000
 23.                2.7200003                                         77.                 2.5699997
 24.                2.3800001                                         78.                 2.6700001
 25.                2.6000004                                         79.                 3.3699999
 26.                2.7700005                                         80.                 2.6899996
 27.                3.1099997                                         81.                 2.2399998
 28.                2.7600002                                         82.                 2.5200005
 29.                2.8500004                                         83.                 2.9399996
 30.                2.8500004
 31.                3.0600004
 32.                2.8400002
 33.                2.8800001
 34.                2.8400002
 35.                2.8500004
 36.                2.8599997
 37.                2.7900000
 38.                2.9600000
 39.                3.0200005
 40.                2.2500000
 41.                3.0799999
 42.                2.2700005
 43.                2.4300003
 44.                2.4600000
 45.                2.6000004
 46.                2.5600004
 47.                2.8400002
 48.                2.8100004
 49.                3.1099997
 50.                2.8699999
 51.                2.7799997
 52.                2.6999998
 53.                2.6099997
 1.     HOMER CITY PLANT UNIT 3 CSA CEM DATA DAILY 1ST OVERLAPPING55.   54.              2.6899996
 2.              2.6300001                                              55.              2.9300003
 3.              2.6000004                                              56.              2.7799997
 4.              2.7500000                                              57.              2.6400003
 5.              2.7700005                                              58.              2.5900002
 6.              2.6599998                                              59.              2.6099997
 7.              2.6099997
 8.              2.7100000
 9.              2.8199997
10.              2.6599998
11.              2.4099998
12.              2.4099998
13.              2.7399998
14.              2.6199999
15.              2.7700005
16.              2.5799999
17.              2.6700001
18.              2.5400000
19.              2.5200005
20.              2.6400003
21.              2.5799999
22.              2.5200005
23.              2.7399998
24.              2.7600002
25.              2.6499996
26.              2.5400000
27.              2.6499996
28.              2.5900002
29.              2.6300001
30.              2.6899996
31.              2.8800001
32.              2.8400002
33.              2.7200003
34.              2.7700005
35.              2.8900003
36.              2.8500004
37.              2.8100004
38.              2.6499996
39.              2.7299995
40.              2.6199999
41.              2.5699997
42.              2.6000004
43.              2.6499996
44.              2.7600002
45.              2.6199999
46.              2.6400003
47.              2.6000004
48.              2.5799999
49.              2.5799999
50.              2.5400000
51.              2.5699997
52.              2.4799996
53.              2.4200001
```

```
                                                                54.            2.4799995
  1.      HOMER CITY PLANT UNIT 3 CSA CEM DATA DAILY 2ND OVERLAPPING55.            2.4499998
  2.                2.4399996                                   56.            2.6099997
  3.                2.5699997                                   57.            2.8000002
  4.                2.7700005                                   58.            2.6400003
  5.                2.7200003                                   59.            2.5299997
  6.                2.4600000                                   60.            2.6199999
  7.                2.5500002                                   61.            2.6499996
  8.                2.5100002                                   62.            2.6300001
  9.                2.5200005                                   63.            2.6700001
 10.                2.5900002                                   64.            2.6000004
 11.                2.7900000                                   65.            2.7500000
 12.                2.6000004                                   66.            2.7100000
 13.                2.5799999                                   67.            2.7299995
 14.                2.7799997                                   68.            2.6300001
 15.                3.0400000                                   69.            2.1000004
 16.                2.4799995                                   70.            2.4499998
 17.                2.4700003                                   71.            2.9300003
 18.                2.5400000                                   72.            2.4200001
 19.                2.7100000                                   73.            2.4700003
 20.                2.5900002                                   74.            2.3900003
 21.                2.8000002                                   75.            2.3299999
 22.                2.8299999                                   76.            2.5299997
 23.                2.6999998                                   77.            2.8100004
 24.                2.5699997                                   78.            2.3299999
 25.                2.6999998                                   79.            2.5000000
 26.                2.5900002                                   80.            2.6300001
 27.                2.6499996                                   81.            2.3999996
 28.                2.4700003                                   82.            2.5799999
 29.                2.5600004                                   83.            2.4799995
 30.                2.5400000                                   84.            2.4499998
 31.                2.5299997                                   85.            2.4200001
 32.                2.4600000                                   86.            2.1000004
 33.                2.4399996                                   87.            2.5100002
 34.                2.3800001                                   88.            2.3199997
 35.                2.4300003                                   89.            2.3999996
 36.                2.5900002                                   90.            2.5500002
 37.                2.5000000                                   91.            2.4499998
 38.                2.7200003                                   92.            2.5799999
 39.                2.5600004                                   93.            2.6000004
 40.                2.6400003                                   94.            2.6499996
 41.                2.8800001                                   95.            2.7399998
 42.                2.4899998                                   96.            2.5400000
 43.                2.4099998                                   97.            2.6599998
 44.                2.4899998                                   98.            2.4399996
 45.                2.5200005                                   99.            2.4200001
 46.                2.4399996                                  100.            2.8900003
 47.                2.0600004                                  101.            2.7600002
 48.                2.3900003                                  102.            2.6899996
 49.                2.3500004                                  103.            2.7200003
 50.                2.3800001                                  104.            2.4799995
 51.                2.7399998                                  105.            2.5100002
 52.                2.5000000
 53.                2.6700001
```

```
106.          2.6599998
107.          2.7299995
108.          2.5299997
109.          2.6199999
110.          2.4899998
111.          2.3999996
112.          2.4399996
113.          2.6199999
114.          2.6499996
115.          2.4300003
116.          2.4399996
117.          2.6400003
118.          2.5699997
119.          2.5400000
120.          2.4499998
121.          2.9099998
122.          2.6099997
123.          2.8599997
124.          2.5600004
125.          2.5299997
126.          2.6099997
127.          2.6000004
128.          2.7700005
129.          2.7100000
130.          2.6499996
131.          2.5900002
132.          2.6700001
133.          2.7799997
134.          2.6400003
135.          2.5400000
136.          2.8699999
137.          2.4700003
138.          2.6400003
139.          2.5500002
140.          2.3400002
141.          2.5699997
142.          2.6000004
143.          2.7299995
144.          2.7200003
145.          2.7299995
146.          2.6999998
147.          2.6000004
148.          2.7500000
149.          2.4700003
150.          2.5900002
151.          2.6000004
152.          2.4600000
153.          2.3900003
154.          2.4600000
155.          2.4300003
156.          2.6099997
157.          2.3199997
```

```
  1.      HOMER CITY PLANT UNIT 3 CSA DATA
  2.                 2.3000002
  3.                 2.2200003
  4.                 2.3299999
  5.                 2.0400000
  6.                 1.8199997
  7.                 2.1599998
  8.                 2.3299999
  9.                 2.6099997
 10.                 2.8299999
 11.                 1.7100000
 12.                 1.9799995
 13.                 2.5200005
 14.                 2.3599997
 15.                 2.3900003
 16.                 1.9200001
 17.                 2.1499996
 18.                 2.2299995
 19.                 2.2100000
 20.                 2.1599998
 21.                 2.2600002
 22.                 2.4600000
 23.                 2.5600004
 24.                 2.7799997
 25.                 2.7900000
 26.                 2.7700005
 27.                 2.4399996
 28.                 2.7100000
 29.                 2.2100000
 30.                 2.3100004
 31.                 2.5699997
 32.                 2.7900000
 33.                 2.4099998
 34.                 2.5600004
 35.                 2.5500002
 36.                 2.7500000
 37.                 2.5900002
 38.                 2.5699997
 39.                 2.7799997
 40.                 2.6300001
 41.                 2.8299998
 42.                 2.6800003
 43.                 2.8199997
 44.                 2.8199997
 45.                 2.5799999
 46.                 2.4799996
 47.                 2.5500002
 48.                 2.6700001
 49.                 2.6999998
 50.                 2.8299999
 51.                 2.5200005
 52.                 2.3299999
 53.                 2.7900000
```